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**MATERIALS DIVISION RESEARCH AND TECHNOLOGY
ACCOMPLISHMENTS FOR FY 89
AND PLANS FOR FY 90**

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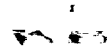
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**MATERIALS DIVISION
RESEARCH AND TECHNOLOGY ACCOMPLISHMENTS FOR FY 89
AND PLANS FOR FY 90**

SUMMARY

The research program of the Materials Division is presented as FY 89 accomplishments and FY 90 plans. The accomplishments for each Branch are highlighted and plans are outlined. Publications of the Division are included by Branch. This material will be useful in program coordination with other government organizations, universities, and industry in areas of mutual interest.

ORGANIZATION

The Langley Research Center is organized by directorates as shown in figure 1. Each directorate is organized into divisions and offices. The Materials Division of the Structures Directorate consists of four branches as shown in figure 2. This figure also shows the technical areas addressed by each Branch. The Division consists of 66 NASA civil servants and 6 members of the Army Aerostructures Directorate, USAARTA, Army Aviation Systems Command located at the Langley Research Center. In addition, about 40 non-personal support contractors work at the Center to add major support to the in-house research program.

FUNCTIONAL STATEMENT

The Materials Division initiates, organizes, and conducts experimental and analytical research on structural materials and their application to aircraft and spacecraft structural systems (figure 3). More specifically the Division:

- Conducts fundamental and applied research studies to develop novel polymeric, metallic, and ceramic materials for advanced structural applications.
- Establishes materials processing and fabrication technology for structural components.
- Demonstrates the application and benefits of advanced materials to specific flight vehicle structures.
- Defines, evaluates, and conducts research on thermal protection materials requirements for high-speed aircraft and space transportation systems.
- Studies the fatigue and fracture behavior of materials to establish practical methods for insuring the structural integrity of aircraft and space structures.
- Characterizes the behavior of structural materials in extreme service environments using test facilities and laboratories for simulation of the flight environment.
- Originates and develops requirements for new facilities and research techniques.
- Operates the mechanics of materials, structural materials, polymer, metallurgical, and environmental effects laboratories.

The long range research thrusts of the Materials Division are shown in figure 4.

FACILITIES

The Materials Division has five major facilities to support its research program.

The Structures and Materials Laboratory houses various environmental effects labs and the metallurgical and metals processing labs. In the environmental effects labs, research is conducted to characterize and enhance the performance of structural materials operating in extreme service environments. Test techniques, instrumentation, and measurement techniques are developed to simulate environmental conditions required to evaluate high-temperature structural materials. The interaction of the space environment on properties of advanced composites, polymer films, and coatings for space systems is studied. Radiation and monoatomic oxygen damage in polymeric materials is studied and chemical formulations for enhanced long-term durability in space are identified.

Fundamental and applied research on advanced metallic and metal-matrix materials is conducted in the metallurgical and metals processing labs. Innovative processing methods for new alloy synthesis and development and fabrication of metallic structural components for future aircraft and space vehicles, including high-temperature applications, are explored. Metallic components are analyzed and tested to demonstrate improvements in advanced metallic alloys and their fabrication processes.

The Mechanics of Materials Laboratory is used to conduct research on the structural integrity of metals and composites for aircraft structures. Tests are conducted to measure the effect of loads on materials under simulated flight conditions. Materials and methods of strength and life prediction for airframes are assessed to develop ways to improve the structural reliability of aircraft.

Fundamental and applied research using advanced polymer synthesis, composites and adhesives processing science, and advanced characterization methodology to develop improved materials concepts for efficient aerospace structures are conducted in the Composites Processing Laboratory. Novel polymeric materials are synthesized for applications such as matrices for fiber-reinforced composites, adhesives for bonding lightweight composite and metal structures, and high-performance films for spacecraft. Innovative processing methods for fabricating composite components for aircraft and spacecraft structures are developed.

Radiation testing of spacecraft materials is conducted in the Space Environmental Effects Laboratory. Spacecraft materials tested include polymeric and metal matrix composites, polymeric films, thermal control coatings, adhesives, solar cells, and laser mirrors.

In addition, the Carbon-Carbon Research Laboratory was completed in July 1988. The Materials Division has expanded its research capability in carbon-carbon materials and this lab houses the processing equipment needed for fabricating carbon-carbon materials and for applying oxidation-protective coatings.

FY 89 ACCOMPLISHMENTS

Polymeric Materials Branch

The Polymeric Materials Branch (figure 5) conducts fundamental and applied research studies combining the disciplines of advanced polymer synthesis, composites and adhesives processing science, and advanced characterization methodology to develop improved materials concepts for efficient aerospace structures. These research and development activities are aimed at achieving maximum structural exploitation of advanced composites and adhesives through development of balanced mechanical/physical properties with good processability. The five year plan for this research is shown in figure 6.

The FY 89 accomplishments of the Polymeric Materials Branch are listed below and are highlighted in figures 7 through 11.

High Performance Polymers

- High Temperature, Low Dielectric Polyimides
- Tensile Film Clips for the Rheovibron
- Polyamic Acid and Polyimide Fibers
- Novel Synthesis of High Performance Heterocyclic Polymers

Composite Processing and Adhesive Bonding

- A Commercially Attractive Thermoplastic Adhesive - Isomer of LaRC-TPI

Mechanics of Materials Branch

The Mechanics of Materials Branch (figure 12) performs research on the integrity of materials for load-bearing structures of metals and composites. This research includes fatigue, fracture mechanics, and structural reliability. Equations and analytical methods are formulated to predict fatigue life and residual strength of damaged and undamaged materials. Design, construction, operation, and inspection methods applied to airframes are assessed to develop ways to improve the overall structural reliability of aircraft and spacecraft. The five year plan of the Branch is shown in figure 13.

The FY 89 accomplishments of the Mechanics of Materials Branch are listed below and are highlighted in figures 14 through 24.

Metals and Metal Matrix Composites

- Effect of Fiber/Matrix Interface Strength in the Mechanical Properties of a Silicon Carbide/Titanium Metal Matrix Composite
- Microstructure/Mechanical Property Relationships for Discontinuous Reinforced Metal Matrix Composites
- Damage to Pressure Vessels by Proof Testing

Composites

- A Mixed-Mode Delamination Test
- Fatigue Life of Stitched Gr/Ep Laminates
- A Micromechanics Treatment of the Effects of Curvature in the Main Load-Carrying Layers on Composite Stiffeners
- Analysis Predicts Onset of Instability-Related Delamination Growth
- Strain Energy Release Rate Analysis of Delamination in a Tapered Laminate Subjected to Tension Load
- Water Intrusion in Thin-Skinned Composite Honeycomb Sandwich Structures

Space Shuttle

- Evaluation of the Resiliency Characteristics of Several Candidate Solid Rocket Booster Elastomeric O-Ring Materials
- Evaluation of O-Ring Gland Surface Finish, Contaminants, and Grease Blockage on the Sealing Performance of the O-Rings During Simulated Space Shuttle Launch Conditions

Applied Materials Branch

The Applied Materials Branch (figure 25) conducts research to characterize and enhance the performance of structural materials operating in extreme service environments. The Branch identifies mechanisms of environmental degradation and failure in structural materials, provides quantitative understanding of degradation mechanisms and evolves models to predict the rate or extent of degradation for various advanced structural materials. Theoretical and experimental studies which relate to the environmental performance of high-temperature materials for thermal protection systems and hot structures of advanced space transportation systems and hypersonic vehicles are conducted. The interaction of the space environment on properties of advanced composites, polymer films, and coatings of interest for space systems is studied. The five year research plan for the Branch is shown in figure 26.

The FY 89 accomplishments of the Applied Materials Branch are listed below and are highlighted in figures 27 through 32.

Space Materials

- Total Absorbed Dose and Dose Rate Effects for Electron Irradiation of Advanced Thermoplastics

Carbon-Carbon Composites

- An Investigation of Fiber Surface Treatment as a Means of Improving Interlaminar Strengths of Carbon-Carbon Composites
- Effect of Surface Machining on Thin 3-D Orthogonal Carbon-Carbon Composites
- Evaluations of Oxidation Resistant Carbon-Carbon Composites in Simulated Hypersonic Vehicle Environments

Composite Materials for Rotorcraft and Aircraft Structures

- Residual Strength of Repaired Graphite/Epoxy Laminates After 5 Years of Outdoor Exposure
- Improved Cure Profiles for Resin Transfer Molded Carbon-Epoxy Composite

Metallic Materials Branch

The Metallic Materials Branch (figure 33) conducts fundamental and applied research studies on advanced metallic and metal-matrix materials. The Branch performs research on advanced high-strength structural alloys and composites to achieve improved mechanical properties through understanding and control of microstructural features. A basic understanding of joining and forming processes for fabricating structural components from advanced metallic materials is developed and innovative processing methods for new alloy synthesis and development and fabrication of metallic structural components for future aircraft and space vehicles are explored. The five year research plan for the Branch is shown in figure 34.

The FY 89 accomplishments for the Metallic Materials Branch are listed below and are highlighted in figures 35 through 40.

Advanced Light Alloy and MMC Development

- Enhanced Diffusion Bonded Ti₃Al-Ti Honeycomb Core Sandwich Panels
- Durability of Graphite/Epoxy Bolted Joint Specimens Demonstrated After 10-Year Exposure Program

Innovative Metals Processing

- Superplastic Forming of Advanced Aluminum Structural Concepts Promise Lighter Weight Structures

High Temperature Thin Gage Metals and MMC for Airframes

- Coatings Improve Performance of Titanium-Aluminides
- High Temperature Aluminum Alloys for Heat Sink Tank Structure
- Development of Titanium Based Metal Matrix Composites for High Temperature Hypersonic Applications

PUBLICATIONS AND PRESENTATIONS

The FY 89 accomplishments of the Materials Division are highlighted by a number of publications and presentations. These are listed by organization and are identified by the categories of formal NASA reports, quick-release technical memorandums, contractor reports, journal articles and other publications, meeting presentations, technical talks, special documents, tech briefs, and patents.

DIVISION OFFICE

Formal Reports

1. Peters, L., Jr.; Dominek, A.; Wozniak, S.; Swann, R. T.; and Hodges, W. T.: NASA LDTM-1048, August 1989.

Quick Release Technical Memorandums

2. Brinkley, K. L.: Materials Division Research and Technology Accomplishments for FY 87 and Plans for FY 88. NASA TM-101506, December 1988, 136 p.
3. Brinkley, K. L.: Materials Division Research and Technology Accomplishments for FY 88 and Plans for FY 89. NASA TM-101593, April 1989, 125 p.
4. Tenney, D. R.; Lisagor, W. B.; and Dixon, S. C.: Materials and Structures for Hypersonic Vehicles. NASA TM-101501, October 1988, 44 p.

Journal Articles and Other Publications

5. Stern, S. A.; Mi, Y.; Yamamoto, H.; and St. Clair, A. K.: Structure/Permeability Relationships of Polyimide Membranes. Applications to the Separation of Gas Mixtures. Journal of Polymer Science: Part B: Polymer Physics, Volume 27, 1989, p. 1887-1909.
6. Tenney, D. R.; and Slemple, W. S.: Radiation Durability of Polymeric Matrix Composites. In American Chemical Society (ACS) Symposium Series - Radiation Effects on Polymeric Materials, E. Reichmanis, J. O'Donnell, eds., 1989, p. 224-251.

Meeting Presentations

7. St. Clair, A. K.; and Stern, S. A.: Structure/Permeability Relationships of Polyimide Films. Presented at the Mid-Hudson Section of the Society of Plastic Engineers, Inc., Third International Conference on Polyimides, November 2-5, 1988, Ellenville, New York. Abstract published in Polyimides: Synthesis, Characterization and Application, p. 215. Proceedings pending.

8. Tullos, G. L.; Cassidy, P. E.; and St. Clair, A. K.: Low Dielectric Polymers Containing the Hexafluoroisopropylidene Group. Presented at the SAMPE 3rd International Electronic Conference, June 20-22, 1989, Los Angeles, California. In Proceedings, p. 219-223.
9. Tullos, G. L.; Cassidy, P. E.; and St. Clair, A. K.: Polymers Derived From Hexafluoroacetone: 12F-Poly(Ether Ketone). Presented at the American Chemical Society Symposium on High Temperature and/or Ladder Polymers, April 9-14, 1989, Dallas, Texas. In Polymeric Materials: Science and Engineering Proceedings, Volume 60, p. 310-315.

Technical Talks

10. Stein, B. A.: Long Duration Exposure Facility - Materials Update. Presented at the Space Station Program Materials and Processes Working Group Meeting, July 11-13, 1989, Kennedy Space Center, Florida.
11. Stein, B. A.: Long Duration Exposure Facility - Materials Special Investigation Group Technical Plan. Presented at the LDEF Investigator Working Group Meeting, May 24, 1989, Williamsburg, Virginia.
12. Stein, B. A.: Materials Research at NASA Langley Research Center. Presented at the Army Materials Science 6.1 Program Review, May 3-4, 1989, Research Triangle Park, North Carolina.
13. Stein, B. A.: Materials Research at NASA Langley Research Center. Presented at the Virginia Commonwealth University Conference on High Technology Material, January 6, 1989, Richmond, Virginia.
14. Stein, B. A.: Space Station Materials Technology at NASA Langley Research Center. Presented at the Space Station Materials and Processes Working Group Meeting, February 14-16, 1989, Huntsville, Alabama.

Patents

15. St. Clair, A. K.; Stoakley, D. M.; and Little, B. D.: Tensile Film Clamps and Mounting Block for the Rheovibron and Autovibron Viscoelastomer. U. S. Patent 4,864,865. Issued September 12, 1989.

Polymeric Materials Branch

Quick-Release Technical Memorandums

16. Gerber, M. K.; Pratt, J. R.; and St. Clair, T. L.: Isomeric Oxydiphthalic Anhydride Polyimides. NASA TM-101525, November 1988, 18 p.

17. Johnston, N. J.; St. Clair, T. L.; Baucom, R. M.; and Towell, T. W.: Polyimide Matrix Composites: Polyimidesulfone/LARC-TPI (1:1) Blend. NASA TM-101568, March 1989, 13 p.
18. Pratt, J. R.; St. Clair, T. L.; Gerber, M. K.; and Gautreaux, C. R.: A Study of Thermal Transitions in a New Semicrystalline Thermoplastic Polyimide. NASA TM-101526, November 1988, 33 p.
19. Progar, D. J.; St. Clair, T. L.; and Pratt, J. R.: Thermoplastic Adhesives Based on a 4,4'-Isophthaloyldiphthalic Anhydride (IDPA). NASA TM-101508, November 1988, 31 p.
20. Tai, H.: Solution of Multi-Center Molecular Integrals of Slater-Type Orbitals. NASA TM-101545, January 1989, 34 p.

Contractor Reports

21. Coguill, S. L.; and Adams, D. F.: Mechanical Properties of Several Neat Polymer Matrix Materials and Unidirectional Carbon Fiber-Reinforced Composites. (NAG1-277 University of Wyoming.) NASA CR-181805, April 1989, 320 p.
22. Hou, T-H.; and Huang, J. Y. Z.: Chemoviscosity Modeling for Thermosetting Resin Systems - IV. (NAS1-18000 Planning Research Corporation.) NASA CR-181807, March 1989, 50 p.
23. Hou, T-H.; Hou, G. J. W.; and Sheen, J. S.: On Processing Development for Fabrication of Fiber Reinforced Composite - II. (NAS1-18000 Planning Research Corporation.) NASA CR-181866, July 1989, 54 p.
24. Stern, S. A.; Vaidyanathan, R.; and Pratt, J. R.: Structure/Permeability Relationships of Silicon-Containing Polyimides. (NAS1-18000 PRC Systems Services.) NASA CR-4237, June 1989, 32 p.
25. Zimmerman, R. S.; and Adams, D. F.: Mechanical Properties of Neat Polymer Matrix Materials and Their Unidirectional Carbon Fiber-Reinforced Composites. (NAG1-277 University of Wyoming.) NASA CR-181631, December 1988, 213 p.

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26. Burks, H. D.; St. Clair, T. L.; and Gautreaux, C. R.: Effect of Thermal History on the Rheological Properties of LARC-TPI. Polyimides: Materials, Chemistry and Characterization, C. Feger, M. M. Khojasteh, J. E. McGrath, eds., Elsevier Science Publishers, 1989, p. 613-624.

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28. Hergenrother, P. M.; and Havens, S. J.: Imide Homo and Copolymers Containing Carbonyl and Ether Connecting Groups. In Polyimides Materials, Chemistry and Characterization, C. Feger, M. M. Khojasteh, J. E. McGrath, eds., Elsevier Science Publishers, 1989, p. 453-463.
29. Hergenrother, P. M.; and Havens, S. J.: Polyimides Containing Carbonyl and Ether Connecting Groups. II. Journal of Polymer Science, Part A: Polymer Chemistry, Volume 27, 1989, p. 1161-1174.
30. Hinkley, J. A.; Crook, R. A.; and Davis, J. R. J.: Chain Dimensions and Rheology of Poly(arylene Ethers). High Performance Polymers, Volume 1, No. 1, 1989, p. 61-71.
31. Hinkley, J. A.; Johnston, N. J.; and O'Brien, T. K.: Interlaminar Fracture Toughness of Thermoplastic Composites. ASTM STP 1044, Advances in Thermoplastic Matrix Composite Materials, G. M. Newaz, ed., 1989, p. 251-263.
32. Hou, T-H.; Bai, J. M.; and St. Clair, T. L.: Semicrystalline Polyimidesulfone Powders. In Polyimides: Materials, Chemistry and Characterization, C. Feger, M. M. Khojasteh, J. E. McGrath, eds., Elsevier Science Publishers, 1989, p. 169-191.
33. Hou, T-H.; Jensen, B. J.; and Bai, J-M: Linear Viscoelastic Properties of a Poly(Arylene Ether Ketone) With Various Molecular Weights. High Performance Polymers, Volume 1, No. 1, 1989, p. 41-59.
34. Hou, T-H.; and St. Clair, T. L.: Characterization of a Semicrystalline Polyimidesulfone Powder. SAE 1988 Transactions, Volume 97, No. 2, September 1989, p. 2.46-2.56.
35. Howes, J. C.; Loos, A. C.; and Hinkley, J. A.: The Effect of Processing on Autohesive Strength Development in Thermoplastic Resins and Composites. ASTM STP 1044, Advances in Thermoplastic Matrix Composite Materials, G. M. Newaz, ed., 1989, p. 33-49.
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39. Pater, R. H.; and Partos, R. D.: New High Performance Semi-Interpenetrating Polyimide Networks and Composites Having Improved Toughness and Microcracking Resistance - Part 4. In Polyimides: Materials, Chemistry and Characterization, C. Feger, M. M. Khojasteh, J. E. McGrath, eds., Elsevier Science Publishers, 1989, p. 37-59.
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55. Hergenrother, P. M.; Beltz, M. W.; and Havens, S. J.: LaRC-CPI, A New Semi-Crystalline Polyimide. Presented at the SAMPE 34th International Symposium and Exhibition, May 8-11, 1989, Reno, Nevada. In Science of Advanced Materials and Process Engineering Series, Volume 34, Book 1, p. 963-975.

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62. Pater, R. H.: Interpenetrating Polymer Network Approach to Tough and Microcracking Resistant High Temperature Polymers - 3. LaRC-RP71. Presented at the 47th Annual Technical Conference and Exhibition of the Society of Plastics Engineers, May 1-4, 1989, New York, New York. In Proceedings, p. 1434-1439.

63. Pater, R. H.: New High Performance Semi-Interpenetrating Polyimide Networks and Composites Have Improved Toughness and Microcracking Resistance. Presented at the Mid-Hudson Section of the Society of Plastic Engineers, Inc., Third International Conference on Polyimides, November 2-5, 1988, Ellenville, New York. Abstract published in Polyimides: Synthesis, Characterization and Application, p. 247. Proceedings pending.
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FY 90 PLANS

Polymeric Materials Branch

Major research thrusts for FY 90 in the Polymeric Materials Branch are in the areas of resin-matrix composite studies, high performance polymers and space durable polymers. The research will be conducted under the following three RTR's.

RTR 505-63-01-01 Resin Matrix Composite Development

Objective:

Develop technology leading to durable, damage tolerant, cost-effective, high performance composites for advanced aerospace structural applications.

Approach:

Scaleup synthesis of promising new polymers and polymer blends and evaluate as composite matrices. Develop and optimize prepreg preparation and composite fabrication. Perform work to develop lower cost prepregging and composite fabrication processes. Test composites under a variety of conditions.

Milestones:

- Correlate damage tolerance of small composite panels with that obtained on standard panels - December 1989.
- 25000 hour aging at 400°F on LARC-TPI, LARC-160 and PMR-15 composites completed - December 1989.
- Determine damage tolerance of composites from one in-house developed high temperature polymer - February 1990.
- Fabricate LARC-TPI tubes using ceramic/high temperature rubber technique - March 1990.
- Scaleup synthesis of one promising new polymer and evaluate in composites - March 1990.
- Optimize parameters on powder impregnation process to make graphite towpreg - May 1990.
- Demonstrate feasibility of preparing uniform thin gage graphite towpreg via powder impregnation - August 1990.
- Clemson University grant will establish method for continuous fusion of polymer powder on carbon tow - September 1990.

RTR 506-43-11-01 High Performance Polymers

Objective:

Develop basic technology leading to high performance and high temperature organic polymers for use as adhesives, composite matrices and films in aerospace applications.

Approach:

Prepare new, high strength, thermally stable, processable polymers and polymer blends (imide, quinoxaline, imidazole, arylene ether ketone, and acetylene containing polymers) and evaluate them as high temperature adhesives, composite matrices and films. Develop fundamental chemical structure/property relationship.

Milestones:

- Improve compression moldability of a semi-crystalline PI through optimization of molecular weight and end-capping - December 1989.
- Prepare and evaluate several novel heterocyclic containing arylene ether polymers - February 1990.
- Prepare new compositions of blends of acetylene containing materials for adhesive and composite evaluation - March 1990.
- Conduct basic synthetic studies on a novel A-B monomer for polyimide formation - April 1990.
- Determine mixing (scrambling) of polyamic acid mixtures - May 1990.
- Complete evaluation of new series of polyimides derived from diamines containing the trifluoromethyl group - July 1990.
- Complete study on imide/arylene ether block copolymers and evaluate as adhesives and composite matrices - August 1990.
- Prepare and evaluate new systems from acetylene terminated compounds and bismaleimides - September 1990.

RTR 506-43-21-05 Space Durable Polymers

Objective:

Develop space-stable, processable, durable composites, adhesives, coatings and films.

Approach:

Prepare new polymers and polymer blends with the chemical structures that impart electron, proton and ultraviolet radiation stability and/or low color. Evaluate these polymers in the form of adhesives, films and coatings in a simulated space environment and monitor their stability through appropriate analytical techniques.

Milestones:

- Novel polyimides containing pendant siloxane segments will be prepared and evaluated in a space environment - December 1989.
- Polyimides containing trifluoromethyl groups will be prepared and evaluated for radiation stability - March 1990.
- Initiate study to determine relationship between polymer chemical structure and stability in a space environment - June 1990.
- Novel siloxane thermosets will be evaluated as space stable coatings and composite matrices - August 1990.

Mechanics of Materials Branch

Research in the Mechanics of Materials Branch will focus on mechanics of damage in laminates and 3-D forms, micromechanics, characterization of the thermomechanical behavior of metal-matrix composites, damage tolerance of light alloys, and computational fracture mechanics. The research will be carried out under the following two RTR's.

RTR 505-63-01-05 Mechanics of Materials Research in Laminated Composites and Metals

Objectives:

Develop the methodology to predict the initiation and growth of critical levels of damage in laminated composites under general mechanical loading conditions. Develop the experimental data base and methodologies to predict the initiation and growth of cracks in metals under constant amplitude and spectrum loading for expected operational conditions. Predict the fatigue and fracture behavior of composites from the fiber, resin, and interface constituent behavior by the application of micromechanics models.

Approach:

- The evolution of damage under general loading conditions will be experimentally observed and documented. The experimental results will be interpreted through appropriate analyses based on mechanics principles. The correlation of the experimental and analytical results will give rise to the development of analytical methods and failure criteria necessary to predict

the strength and life of composite structures. Failure methodology will be verified by predicting the behavior of tapered laminates subjected to combined loads typical of those experienced in composite rotor hub flexure.

- Experimentally evaluate the fatigue and fracture performance of promising new alloys relative to established alloys and evaluate the adequacy of current methods to predict the life of new alloys under expected spectrum loading and temperature conditions. Broaden the applicability of current life-prediction methods by developing methods to describe mixed-mode crack-growth behavior and by developing a three-dimensional model of closure incorporating both plasticity- and roughness-induced closure behavior. Develop the fracture mechanics methodology and generate solutions to cracked-body boundary value problems consistent with the applied loads and service environment for launch vehicles and supersonic transport vehicles. Establish correlations between observed performance and metallurgical features which might be used to guide development of alloys with improved performance.
- Conduct tests to identify microdamage onset and growth and to determine inelastic constitutive relationships that account for multiaxial stress states and microdamage. Then use the constitutive relationships in composite stress analyses to compute stresses, strains, and fracture mechanics parameters corresponding to observed composite failures. Establish constituent failure criteria by comparing the observed microfailures with computed stress analysis results. Finally, develop models to relate composite failures to the corresponding fiber, matrix, and interface constituent failures.

Milestones:

- Complete elastic and elastic-plastic analyses of surface cracks in welded aluminum plates under tension and bending loads and compare with experimental displacement measurements - June 1990.
- Extend the plasticity induced crack-closure model to include first order time and temperature dependent effects and load interaction under cyclic loading - September 1990.
- Develop strain energy release rate expressions for delaminations growing from matrix cracks and discontinuities in laminated composites to support the implementation of a progressive failure model for strength and life predictions - September 1990.
- Conduct an experimental and analytical investigation of the effects of in-plane loads on the development of damage during a foreign object impact - September 1990.

RTR 506-43-71-03 Mechanics of Damage in Metal Matrix Composites

Objectives:

Develop the methodology necessary to predict the fatigue, fracture, and mechanical behavior of MMC's required to insure structural integrity at elevated temperatures in support of hypersonic vehicles.

Approach:

The mechanical behavior of MMC's and the development of damage mechanisms under thermal and mechanical loadings will be experimentally observed and documented. Material models and mechanics analyses will be developed to explain the observed material behavior. These analyses will include the effects of plasticity, viscoelasticity, and thermal stresses. The correlation of the experimental and analytical results will give rise to the development of analytical methods and failure criteria necessary to predict the strength and life of laminated composite structures.

Milestones:

- Establish relationship between the microstructure of discontinuous MMC's and the global mechanical properties using micromechanics - March 1990.
- Conduct thermomechanical fatigue tests on the SiC/Ti MMC to quantify the potential benefits in life achieved by coating the SiC fibers with TiB₂ - September 1990.

Applied Materials Branch

Research emphasis in the Applied Materials Branch for FY 90 will be in the areas of space materials, carbon-carbon composites, and composite materials for aircraft and rotorcraft structures. This research will be carried out under the following five RTR's.

RTR 505-63-01-06 Advanced Composite Materials and Processes

Objectives:

To develop and demonstrate the potential of innovative damage tolerant composite materials and affordable processes for applications to aircraft structures.

Approach:

In-house, contractual, and grant studies will be conducted to develop damage-tolerant materials and processing science concepts for composite aircraft structural applications. Composite materials that incorporate toughened resins and high strength/strain fibers will be evaluated for improved damage tolerance. New net-shaped material forms that are fabricated with automated textile processes such as 2-D and 3-D weaving, stitching/knitting and braiding

will also be evaluated. The focus will be on improved damage tolerance and affordable RTM techniques. Process models to predict resin infiltration through textile preforms will be developed. Sensors will be used to aid in processing and validating predicted infiltration. Analytical models to predict the mechanical response and damage tolerance of textile composite materials will be developed and verified. Long-term durability of composites in service and repaired composite components will be established.

Milestones:

- Conduct mechanical property and damage tolerance tests on knitted/stitched material - December 1989.
- Complete 8.5 year flight service evaluation of composite components on S-76 and publish final report - December 1989.
- Complete 15-year and final residual strength tests on B-737 graphite/epoxy spoilers - December 1989.
- Conduct 1/4 lifetime spectrum fatigue on repaired graphite/epoxy components after 6 years of outdoor exposure at NASA Langley - December 1989.
- Conduct shear tests of RTM processed hat-stiffened and T-stiffened graphite/epoxy panels - March 1990.
- Complete testing of composite components after 7 years of flight service on Bell 206L helicopters - June 1990.
- Complete study on use of dielectric monitor for on-line feedback for curing of RTM composites - June 1990.
- Complete RTM and initiate testing of woven panels with through-the-thickness fiber reinforcements - June 1990.
- Evaluate the mechanical properties and damage tolerance of new toughened matrix composite materials - June 1990.
- Evaluate the effect of cyclic loading on impact damaged composite material - June 1990.
- Complete verification of RTM process model for optimizing RTM processing conditions - September 1990.
- Evaluate the software requirements for integration of the dielectric monitor and RTM process model for automated control of RTM composites - September 1990.

- Conduct material characterization tests on filament wound graphite/thermoplastic materials - September 1990.
- Complete evaluation to determine the effect of fill yarn compliance on compression strength of uni-woven RTM panels - September 1990.
- Conduct analytical trade studies to evaluate reinforcing concepts for improved impact-resistant designs - September 1990.

RTR 506-43-21-04 - Composite Materials for Spacecraft Applications

Objectives:

Develop the technology base for high performance, environmentally and dimensionally stable composite tubes, reflector panels, adhesives, and coatings for use in spacecraft structures.

Approach:

Exploit advances in fiber and matrix material technology to develop new high performance composite materials. Develop and evaluate adhesives for tube end-fitting joints and sandwich construction reflector panels. Develop and evaluate polymeric films for space structural applications. Develop facilities for combined space environmental exposures. Evaluate materials performance in simulated long-term Earth orbiting environments. Develop analytical models to guide development of new materials and predict mechanical and thermal response. Verify models and ground simulations with available flight data.

Milestones:

- Determine the shear properties of P75/930 composites as a function of total electron radiation dose - December 1989.
- Initiate study on effects of electron radiation and atomic oxygen on LaRC-TPI resin - March 1990.
- Determine effects of Gr fiber modulus on microdamage/thermal expansion during thermal cycling - June 1990.
- Determine feasibility of incorporating organometallic and organosilicon compounds into polymers for increased atomic oxygen resistance - June 1990.
- Determine atomic oxygen, UV, and electron radiation stability of transparent polymeric films - June 1990.
- Determine the effects of long-term thermal cycling combined with electron radiation on composite tubes - September 1990.

- Develop techniques for cure-optimization of adhesives using dielectric properties - September 1990.
- Complete study on effects of electron radiation and thermal cycling on composite joints bonded with epoxy adhesives - September 1990.
- Fabricate (in-house) and evaluate the thermal cycling stability of a ultra-high modulus (50 Msi) composite tube - September 1990.
- Complete initial evaluation of MD LDEF specimens - September 1990.

RTR 506-43-71-02 Carbon-Carbon Composites for Generic Hypersonics

Objectives:

Develop high strength, minimum gauge, oxidation-protected carbon-carbon materials for hot structural applications on hypersonic vehicles.

Approach:

Advanced processing methods, fiber surface modifications, and alternate reinforcement concepts will be developed to improve substrate mechanical properties and the compatibility of substrates with oxidation-resistant coatings. The potential benefits of alternate matrix precursor materials and advanced fibers will also be explored. Matrix and fiber oxidation inhibitors, sealants, and advanced coatings will be developed to improve oxidation resistance. Environmental testing will be performed in simulated mission dynamic environments and in multiparameter (temperature, pressure, load) facilities.

Milestones:

- Potential assessed for conversion coating 3-D orthogonal carbon-carbon composites with both machined (smoothed) and unmachined surfaces - March 1990.
- Potential assessed for improving conversion coatability of 2-D carbon-carbon by using small-diameter-tow reinforcement - June 1990.
- Conduct feasibility studies of sol-gel-prepared overcoats on conversion coated C-C substrates - June 1990.
- Molding parameters for phenolic-based carbon-carbon composites defined for improved interlaminar properties - September 1990.

RTR 585-02-21-01 Advanced Materials for PSR

Objectives:

Develop advanced composite materials and coatings that are durable and have stable thermal and mechanical properties in the space service environment of precision segmented reflector spacecraft.

Approach:

New, novel polymer resins will be developed and used to fabricate composites. These composites and existing alternate composite materials will be developed and used to fabricate honeycomb panels. Alternate composite fabrication methods that result in lower residual stresses will be investigated. Material constitutive equations and analytical models will be developed to correlate/predict environmental effects on thermal and mechanical properties of the advanced composites. These models will aid in directing the materials development activities. The surface distortion of composite laminates/panels before and after thermal cycling will be measured and modeled.

Milestones:

- Parametric study of effects of constituent properties on composite/honeycomb panel response complete - December 1989.
- Upgrade IR interferometer dilatometer to measure 10" panels - March 1990.
- Fabricate parabolic PSR panel with alternate composite material facesheets - March 1990.
- Viscoelastic analysis of honeycomb panels thermal response complete - June 1990.
- Determine properties of low temperature cured T300/934 - June 1990.

RTR 763-01-41-17 Oxidation-Resistant C-C Composites for NASP

Objectives:

Develop oxidatively protected carbon-carbon material concepts to meet airframe requirements in support of National Aero-Space Plane.

Approach:

Evaluate in simulated NASP mission environments various promising oxidation-protection systems which were developed for propulsion applications. Build on these results, tailoring a new oxidation-protection system (in-depth oxidation protection, sealants, coatings) to meet specific NASP mission requirements.

Milestones:

- Continue conducting mechanical property and mission simulation performance testing on Rohr and BFG candidate materials - Continuing.
- Initiate arc jet tests of Rohr and BFG candidate materials - December 1989.
- Conclude LTV field-sealant development contract, take delivery of sealants and test specimens, and initiate in-house evaluations - December 1989.
- Award multiple contracts for improving adherence of coatings on carbon-carbon composites in NASP environments - March 1990.
- Take delivery of HITCO candidate oxidation-resistant carbon-carbon (ORCC) material and initiate performance evaluations in multiparameter facility and arc jets - March 1990.
- Initiate mission simulation tests on ORCC composites in support of NASP MASAP Consortium Team on Refractory Composites - June 1990.
- Complete installation of improved computer control system for multiparameter facility - September 1990.

Metallic Materials Branch

Research in the Metallic Materials Branch for FY 90 will focus on advanced light alloy and metal-matrix composites development, innovative metals processing, and high temperature, thin gage metals and metal-matrix composites for airframe applications. This research will be carried out under the following five RTR's.

RTR 505-63-01-02 Advanced Structural Metallics for Service to 1000°F

Objectives:

To develop a fundamental understanding of the metallurgical structure/mechanical property interactions resulting from powder processing, consolidation, and subsequent thermomechanical processing of intermediate and high temperature aluminum alloys prepared by advanced ingot and powder metallurgy techniques. To demonstrate the property and durability advantages of advanced aluminum alloys for aerospace structures. To develop advanced inorganic composite materials for aerospace structural applications.

Approach:

Prepare new aluminum alloy compositions of laboratory quantities by advanced I/M and P/M techniques. Develop and evaluate promising in-situ composite materials systems with light alloy metallic matrices and correlate microstructural/mechanical property relationships. Identify metallurgical

characteristics controlling specific properties through laboratory analysis and development of optimized processing techniques to obtain tailored properties.

Milestones:

- Develop environmental degradation laboratory capabilities to support salt spray testing and alternating service atmosphere exposure - October 1989.
- Evaluate the electrochemical response and stress corrosion cracking resistance of weldalite - December 1989.
- Determine consolidation and fabrication practices for Mn, Ca, Si containing, low density, high-temperature aluminum alloys produced from powder atomized at the LaRC gas atomization facility - January 1990.
- Complete system design study for a reusable space launch booster for the advanced launch system program - March 1990.
- Complete mechanical property evaluation of improved mechanically alloyed material Al905XL - March 1990.
- Complete initial evaluation of high-temperature aluminum alloy candidate materials for advanced launch systems - May 1990.
- Determine environmental factors which contribute to accelerated corrosion fatigue of 2024-T3 - June 1990.
- Determine the effect of thermal exposures associated with welding and forming on microstructure and properties of dispersion strengthened aluminum alloys - August 1990.
- Determine the effects of various Cu-Li ratios on the microstructure and mechanical properties of Al-Cu-Li-In-Zr aluminum alloy variants heat-treated to the T6 condition - September 1990.

RTR 505-63-01-03 Innovative Metals Processing

Objectives:

Develop improved aluminum alloys and innovative processing methods for fabricating lightweight aerospace structural components. Develop advanced forming and joining techniques for lightweight Al-Li and high temperature aluminum alloys and evacuated titanium honeycomb-core sandwich concepts.

Approach:

Combined in-house, contractual, and university efforts to define the potential of advanced aluminum alloys for aerospace structural applications. Demonstrate weldability, enhanced post-SPF properties and evaluate the cryogenic

behavior of superplastic Al-Li alloys of modified compositions. Assess the potential of high temperature aluminum alloys and develop improved brazing and joining processes for fabricating structural subelements. Characterize material properties and design, fabricate, and test structural elements.

Milestones:

- Establish resistance spot welding parameters for Al-Li alloy 2090 and 8090 - November 1989.
- Determine SPF characteristics of weldalite - March 1990.
- Superplastically form 2090 + In isogrid stiffeners for cryogenic tank - May 1990.
- Complete testing of SPF 7475 and 8090 Al hat stiffened crippling panels for cryogenic applications - November 1990.
- Fabricate and test SPF column buckling panels - November 1990.
- Design of full thickness cryogenic tank compression panels having panel joints - December 1990.

RTR 505-80-31-02 Advanced Metal Matrix Composites

Objectives:

Develop specific, high temperature metal matrix composites and associated fabrication technology for NASP applications.

Approach:

Establish surface treatments/coatings for selected fiber/matrix stability. Fabricate and test composite laminates to establish performance limits. Define scale-up requirements for large panel manufacture.

Milestones:

- Publish report on Task I results - November 1989.
- Fabricate multi-layer, cross-ply laminates - October 1990.

RTR 506-43-71-01 High Temperature Metallics for Generic Hypersonics

Objectives:

Develop new high temperature materials and associated processing and joining techniques for use up to 2000°F including alloys, light alloy intermetallics, compatible reinforcing phases and metal matrix composites.

Approach:

Combined in-house and contract research to develop and characterize advanced metallic systems produced by deposition techniques, rapid solidification rate technology and conventional processing. Establish joining practice for very thin gage, lightly loaded structures. Develop compatible fibers/coatings for stable, high temperature metal composites.

Milestones:

- Evaluate thin gage Ti_xAl and Be substrates produced by PVD - December 1989.
- Demonstrate use of EDB processing to fabricate high quality IM $Ti_3Al/Ti-15-3$ honeycomb sandwich panels - February 1990.
- Initiate program to develop new fibers/compliant layer coatings for specific composite systems - March 1990.
- Determine compatibility of new high temperature and oxidation resistant titanium alloys with state-of-the-art reinforcements - May 1990.
- Demonstrate feasibility of PVD techniques to fabricate thin reinforced intermetallics - September 1990.

RTR 763-01-41-11 Ti_xAl Composites for NASP

Objectives:

Develop advanced joining processes for fabricating Ti_xAl metal-matrix composite (MMC)/honeycomb core sandwich structure, assess direct powder metallurgy consolidation of Ti_xAl MMC, and develop an analytical model for prediction of composite fatigue behavior.

Approach:

Conduct studies using available titanium based materials to develop joining processes suitable for fabricating Ti_xAl MMC/honeycomb core sandwich structure. Assess candidate processes including enhanced diffusion bonding (EDB) based on metallurgical studies and mechanical property tests. Evaluate alternate EDB material compositions and process parameters to improve elevated temperature properties of joints between facesheets and core. Fabricate, test and evaluate small sandwich specimens and structural sub-elements using Ti_xAl MMC's as they become available. Develop direct powder metallurgy(PM) consolidation process for manufacture of SiC/Ti_xAl MMC using blended matrix powders and engineered fiber/matrix interface. Evaluate feasibility of this process using microstructural evaluation, mechanical testing and fracture analysis of consolidated laminates.

Milestones:

- Complete flatwise tension and edgewise compression tests of EDB IM Ti₃Al/Ti-3-2.5 H/C sandwich specimens at room and elevated temperatures - December 1989.
- Modify micromechanics model of continuous fiber reinforced MMC to include thermoviscoplasticity analysis - January 1990.
- Demonstrate feasibility of direct PM consolidation of SiC fiber reinforced Ti_xAl matrix composites using blended powders and engineered fiber/matrix interfaces - March 1990.
- Evaluate thermo-mechanical fatigue behavior of SCS-6/Ti-15-3 MMC - April 1990.
- Evaluate EDB IM Ti₃Al/Ti-15-3 H/C sandwich panels - April 1990.

Concluding Remarks

This document presents the FY 89 accomplishments, presentations and publications, and the FY 90 research plans of the Materials Division.

LANGLEY RESEARCH CENTER

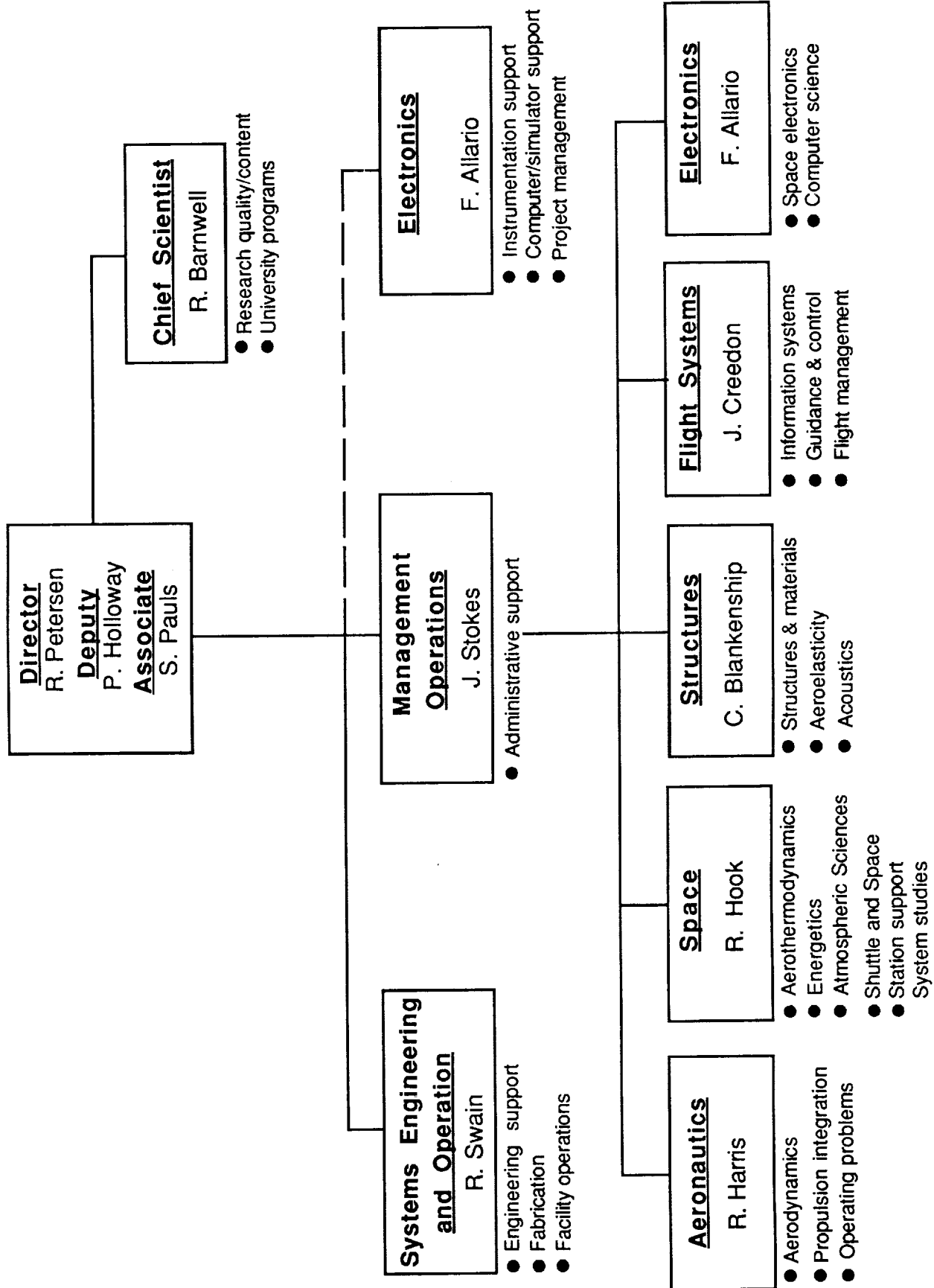
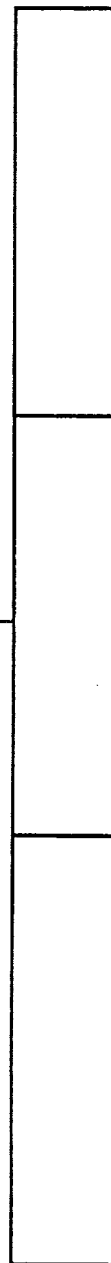


Figure 1.

MATERIALS DIVISION

Darrel Tenney, Chief
Bland Stein, Assistant Chief
Shirley Crockett, Secretary



Polymeric Materials Branch
Terry St. Clair

- High-performance polymers
- Polymer charact.
- Tough composites

Metallic Materials Branch
Barry Lisagor

- Light alloy MMC development
- Innovative metals processing
- High temp. thin gage metallics

Applied Materials Branch
Howard Maahs

- Environmental effects
- Thermomech. stability
- Carbon-carbon
- Advanced composite material concepts

Mechanics of Materials Branch
Charles Harris

- Micromechanics of delamination
- Fat. & Fract. of metals
- Fat. & Fract. of MMC

Figure 2.

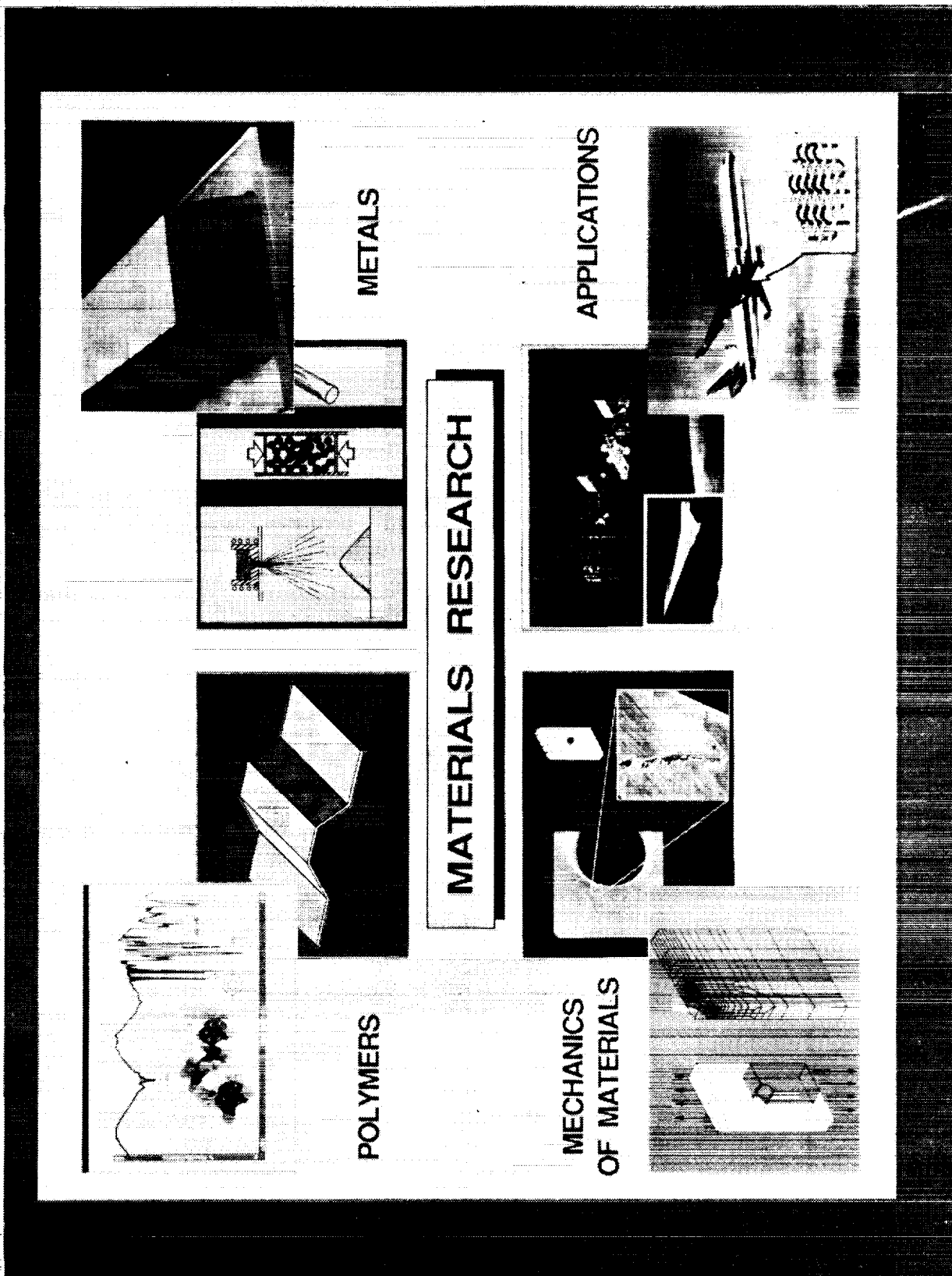


Figure 3.

MATERIALS DIVISION

LONG RANGE THRUSTS - AERONAUTICS

Lead Role

- Metallic materials for aircraft structures
- Carbon-carbon composites for hypersonic vehicles
- High temperature composites and Al alloys for high speed aircraft

Support Role

- Composite materials for primary aircraft structures

Figure 4(a).

MATERIALS DIVISION

LONG RANGE THRUSTS - SPACE

Lead Role

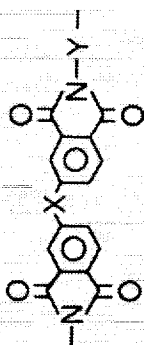
- Materials & structures technology for Advanced Launch Systems
- Materials durability in the space environment

Support Role

- Structures, materials and dynamics technology for Space Station

POLYMERIC MATERIALS

Novel synthesis



Matrix resins Adhesives Films

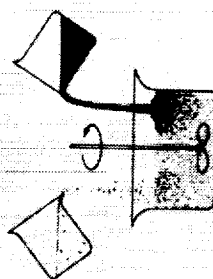
Advanced characterization



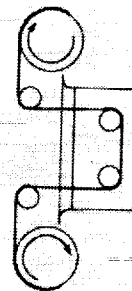
Chemical, physical & mechanical

Structure
property
relationships

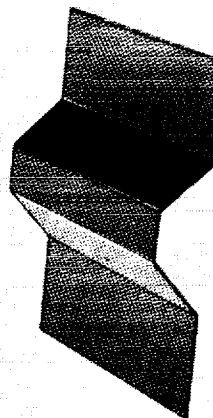
Composite development



Resin development



Fiber impregnation



Composite processing

Figure 5.

POLYMERIC MATERIALS BRANCH
FIVE YEAR PLAN

MAJOR THRUST	FY89	FY90	FY91	FY92	FY93	EXPECTED RESULTS
High performance polymer concepts						New polymers and polymer technology
				Develop polymer structure/property relationships		
				Synthesis of novel monomers and polymers		
				Polymer additives/fillers		
Composite matrices				Improved polymer characterization technology		Better understanding of high performance materials
				Siloxane/inorganic polymers		
				Constituent property/composite property relationships		
				Thermoplastics (TP), TP/thermoset blends & semi-cryst. polymers		
Composite processing and adhesive bonding				Innovative prepreg development		Balanced composite properties
				New composite processing concepts (powder, hot melt, etc.)		
				Adhesives development and characterization		
						Advanced processing and bonding technology for high performance materials

Figure 6.

HIGH TEMPERATURE, LOW DIELECTRIC POLYIMIDES

Anne K. St. Clair
Materials Division
Ext. 44242 November 1988
RTOP 505-63-91
Code RM WBS 52-2

Research Objectives: (1) Gain a basic understanding of the mechanisms which affect the dielectric constant of linear aromatic polyimides, and (2) synthesize and develop high temperature, low dielectric polyimides for aerospace applications.

Approach: Conduct a structure/property relationship study to determine how variation in molecular structure of an aromatic polyimide affects the dielectric constant. Characterize neat resins, films, coatings and composites and compare properties with those of state-of-the-art materials.

Accomplishments: An extensive structure/property relationship study involving approximately 100 polymers has resulted in numerous linear polyimides which exhibit low dielectric constants in the range of 2.4 - 2.8. A sampling of polyimides prepared from the hexafluoropropane-containing dianhydride 6FDA are shown in Fig. 7(b). Low dielectric constants have been achieved by altering the molecular structure of the polymer to reduce or eliminate interactions between polymer chains and by the incorporation of fluorine-containing groups [Fig. 7(c)]. The resulting high temperature polymers are easily processed to form low dielectric films, coatings and fiber-reinforced composites. Films of this study are significantly more resistant to moisture than commercial polyimide film.

Significance: The dielectric constant of the commercial polyimide presently used as the state-of-the-art passivant and interlevel dielectric for electronic applications ranges from 3.2 to 4.0 depending upon moisture content. The lower limit of 3.2 is obtained only after full desiccation. As such a film or coating absorbs moisture, the dielectric constant rises making measurements difficult and greatly hindering the operation of electronic devices. The low dielectric polyimides of this study are highly suitable film, coating and composite matrix materials for both aerospace and industrial applications where thermal stability, high electrical insulation and resistance to moisture are required.

Future Plans: Basic research to improve understanding of mechanisms for lowering the dielectric constant of high performance polymers will continue along with the synthesis of new, low dielectric resins. Three U.S. companies are currently negotiating with NASA to license this technology and have plans to make several polymer systems commercially available in the foreseeable future.

Figure 7(a).

DIELECTRIC PROPERTIES OF POLYMERS PREPARED FROM 6FDA DIANHYDRIDE



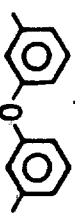
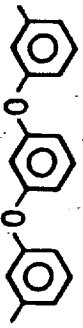

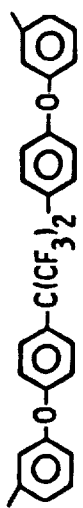
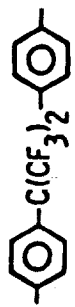
Polymer	Diamine Ar	Dielectric constant at 10 GHz
6FDA + DDSO ₂		2.86
6FDA + 4,4'-ODA		2.79
6FDA + 3,3'-ODA		2.73
6FDA + APB		2.67
6FDA + 4-BDAF		2.50
6FDA + 3-BDAF		2.40
6FDA + 4,4'-6F		2.39

Figure 7(b).

LOW DIELECTRIC POLYIMIDES

(1) The dielectric constant of a linear aromatic polyimide can be lowered by incorporation of the following into the polymer molecular structure:

- Separator groups ($\overset{\text{O}}{\parallel}$, $-\text{O}-$, $-\text{S}-$, $-\text{C}-$, etc)
- Physical "kinks" (meta isomerism)
- Large, bulky groups ($-\text{SO}_2-$, $-\text{CF}_3-$)
- Fluorine-containing groups ($-\text{CF}_3$)

(2) Low DE polyimides of this study have the following properties:

- Improved resistance to moisture
- Ease in processing (films and composites)
- Excellent thermal stability and mechanical strength

Figure 7(c).

TENSILE FILM CLAMPS FOR THE RHEOVIBRON

Diane M. Stoakley and Anne K. St. Clair

Polymeric Materials Branch
Ext. 44246 January 1989
RTOP 505-63-91
Code RM WBS 52-2

Research Objective: Design film clamps that reduce data variability for the Rheovibron, a commercially available instrument for determining glass transition temperature, tensile modulus and damping of thin polymer films. This reduced variability was necessary to enable polymer structure-property relationship studies to be conducted on the Rheovibron.

Approach: Design and construct tensile film clamps and film mounting block that provide uniform sample gripping and improved alignment in the instrument. The design would eliminate major sources of error that had previously prevented quantitative thin film analysis on this instrument.

Accomplishments: Tensile clamps were designed that consisted of a "T" and "U" clamping assembly. (See Figure) Thin polymer film specimens were mounted in the T-clamps using a mounting fixture that was removed from the Rheovibron. The T-clamps and film were then positioned into U-clamps. The U-clamps were connected to the instrument stress and strain gauges and left in place at all times. This clamping arrangement provided reproducible sample mounting and minimized operator dependence.

Data were obtained on DuPont Kapton H film using the standard instrument clamps and the newly designed T and U-clamps. The new clamps (See Table) provided significantly reduced data variability and more accurate modulus values.

Significance: The new tensile film clamps provide reproducible sample mounting and gripping. The reduced data variability achieved by use of these clamps will allow polymer structure-property relationship studies in thin films, previously unattainable because of the large variability in modulus values obtained with the standard clamps.

Future Plans: A patent application is being filed on this invention. This technology should prove attractive to designers and manufacturers of instrumentation for measuring dynamic mechanical property of thin films.

Figure 8(a).

NEWLY DESIGNED RHEOVIBRON

TENSILE CLAMPS

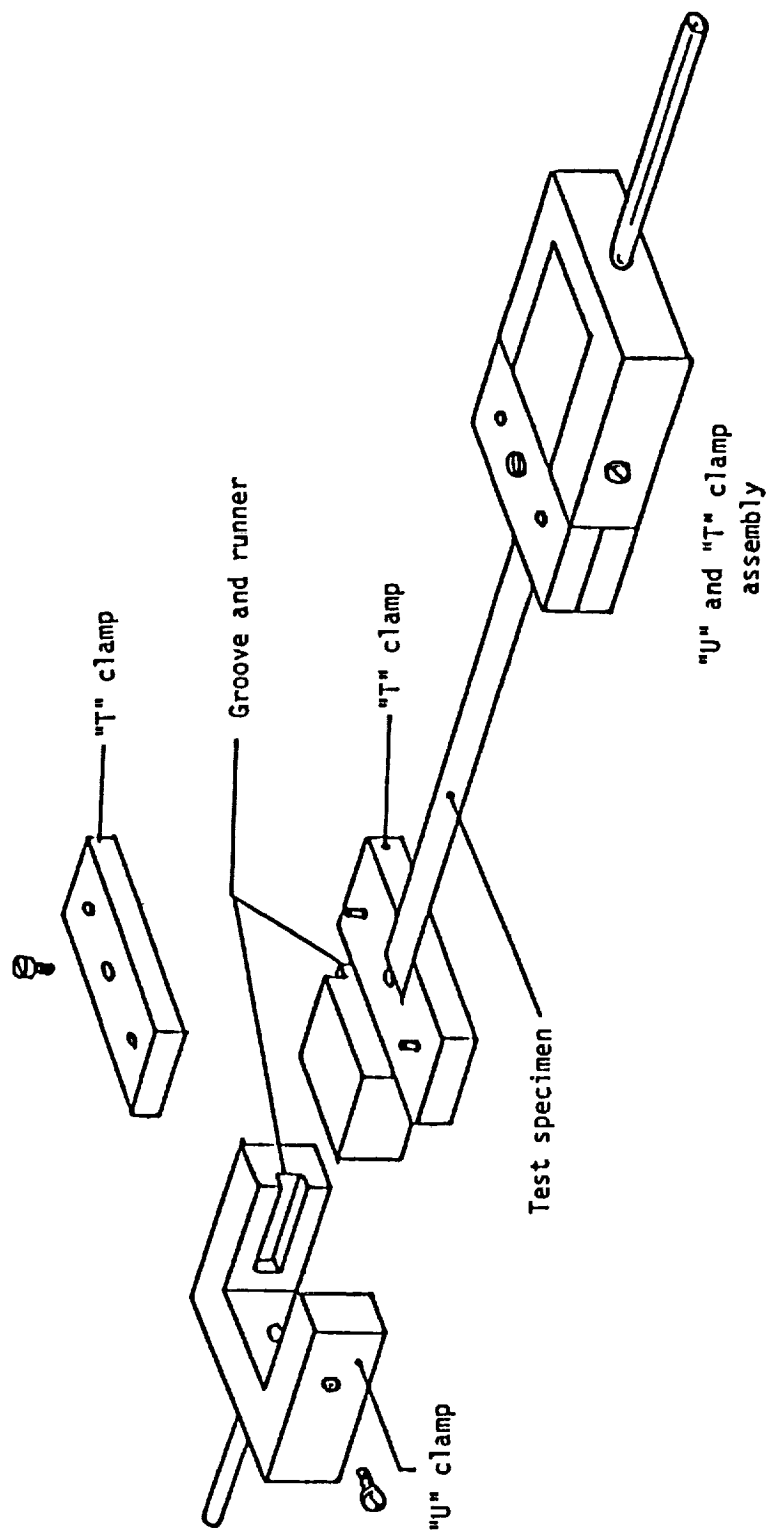


Figure 8(b).

NEW CLAMPS PROVIDE REDUCED DATA VARIABILITY

Run #	Tensile Modulus, psi*	
	Standard clamps	NASA clamps
1	537,000	443,000
2	572,000	441,000
3	459,000	440,000
4	519,000	442,000
Average	522,000	442,000
	6.3% deviation from mean	0.3% deviation from mean

*Dupont® literature value 430,000 psi

Figure 8(c).

POLYAMIC ACID AND POLYIMIDE FIBERS

Anne K. St. Clair and William E. Dorogy, Jr.

Materials Division

Ext. 44242 May 1989

RTOP 505-63-91

Code RM WBS 52-2

Research Objective: (1) Produce solid polyamic acid fibers by extruding polymer resin solutions into a liquid coagulation medium that induces filament formation and (2) convert the polyamic acid fibers into their polyimide form.

Approach: Extrude the polyamic acid resins into a variety of liquid coagulation mediums under different conditions. Alter the following variables to produce a solid filament as it exits the coagulation medium: coagulation medium composition and concentration, filament diameter, inherent viscosity, % solids and type of resin. Analyze the void content of the fibers using scanning electron microscopy and determine the fiber tensile properties.

Accomplishments: Relationships exist between the production of solid polyamic acid filaments and the coagulation medium composition and concentration, filament diameter, inherent viscosity, % solids and type of resin. Solid filaments have been produced using a N,N-dimethylacetamide solution of a polymer prepared from 3,3',4,4'-benzophenonetetracarboxylic dianhydride and 4,4'-oxydianiline at 15% solids with a minimum inherent viscosity of 1.6 dl/g (see figure). Suitable coagulation mediums included 70 - 75% aqueous ethylene glycol or 70 - 80% aqueous ethanol at a temperature of 20°C. Filament diameters generally had to be kept below 50 microns. However, increasing the resin inherent viscosity substantially above 1.6 dl/g allowed solid fibers with filament diameters in excess of 50 microns to be produced. Thermal conversion of the polyamic acid fibers to their polyimide form was achieved by heating for one hour at each 100°, 200° and 300°C. Tenacity, initial modulus, yield point and % elongation for the solid polyimide fibers were greater than the corresponding values for films and non-solid fibers produced using this resin, as is shown in the accompanying figure. Tensile properties of these solid polyimide fibers were comparable to standard textile values.

Significance: Polyimide fibers can provide the high temperature stability and excellent chemical resistance for which this class of polymers is noted. It is necessary, but difficult however, to obtain solid core fibers from polyamic acid resins. This research has resulted in spinning conditions that enable the production of solid core fibers and the accompanying increase in mechanical properties.

Future Plans: Basic research is needed to investigate the use of other coagulation medium compositions and concentrations, to improve the tensile properties of both the polyamic acid and resulting polyimide fibers and to produce polyimide fibers directly from resins consisting of a soluble polyimide dissolved in a suitable solvent.

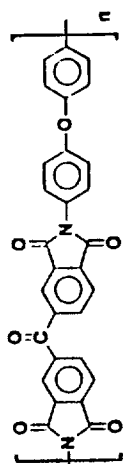
Figure 9(a).

WET SPINNING OF POLYAMIC ACID FIBERS

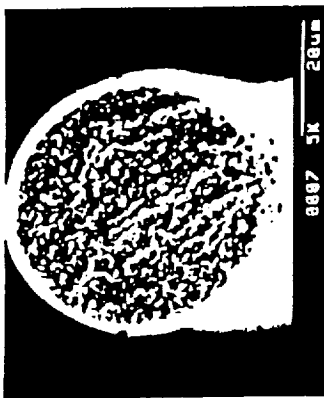


Extruded into

60% Aqueous Ethanol



Polyimide



Extruded into

70% Aqueous Ethanol

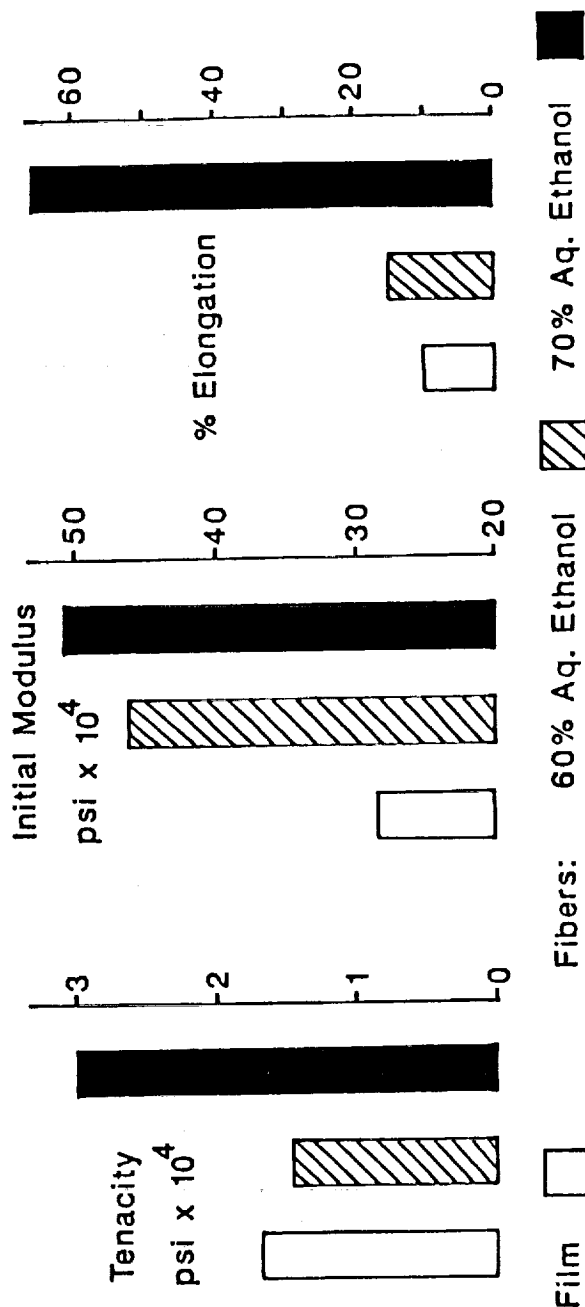


Figure 9(b).

NOVEL SYNTHESIS OF HIGH PERFORMANCE HETEROCYCLIC POLYMERS

John W. Connell and Paul M. Hergenrother
Polymeric Materials Branch
Ext. 44264 July 1989
RTOP 506-43-11
Code RM WBS 52-2

Research Objective: Develop a new, lower cost, easier synthetic route to high performance/high temperature heteroaromatic polymers for potential use as films, coatings, adhesives and composite matrices on aerospace structures and vehicles.

Approach: Prepare polymers by reacting novel bisphenol heterocyclic monomers with activated aromatic dihalides.

Accomplishments: Several polyphenylquinoxalines (PPQs) have been prepared by this new route which avoids the expensive bis(phenyl-a-diketone) monomers used in conventional PPQ preparation. In addition, this approach allows for the preparation of semi-crystalline PPQs which were previously unknown.

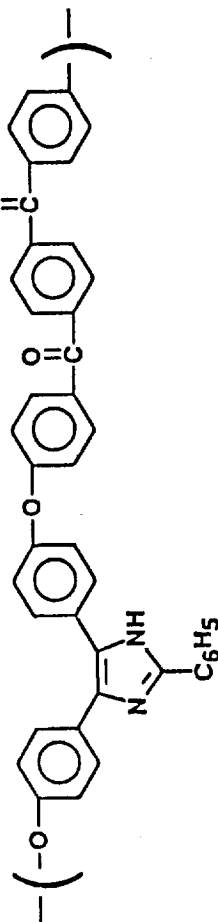
Novel polyimidazoles were also prepared utilizing this approach that had been unattainable in high molecular weights by other synthetic routes. The properties of a representative polyimidazole are presented in the accompanying table. Of particular note are the excellent fracture toughness and adhesive properties. Ti/Ti tensile shear specimens retained high strength when tested at 200°C after aging 500 hours at 200°C.

Significance: This synthetic route is applicable to the preparation of a wide variety of poly(aromatic heterocyclic) materials. In many cases a combination of advantages including cost effectiveness, unique chemical structures and facile chemical structure variation are realized. Polymers prepared via this unique route are potentially useful as adhesives, composite matrices, coatings, films, membranes and moldings.

Future Plans: This novel approach will be expanded to include other families of heterocyclic polymers in order to obtain unique combinations of properties to fill varying needs of the aerospace industry. In addition, more extensive composite and adhesive work will be performed on the polyimidazoles.

Figure 10(a).

Polyimidazole Properties.



Glass Transition Temperature: 243°C

Fracture Energy (G_{IC})*: 22.9 in lb/in²

Thin Film Properties

Test Temperature, °C	25	177	200
Tensile Strength, Ksi	14.2	8.2	6.6
Tensile Modulus, Ksi	407.0	306	273
Elongation, %	6.0	6.0	7.5

II/II Tensile Shear Properties*

Test Temperature, °C	25	177	200
Strength, Ksi	4.8	3.8	3.1 (3.5)**

**After 500 hrs at 200°C

Unidirectional AS-4 Laminate Properties*

Test Temperature, °C	25	150
Flexural Strength, Ksi	240	144
Flexural Modulus, Ksi	17.6	12.5
Short Beam Shear Strength, Ksi	9.1	7.5

*Compression Molding Conditions 300°C under 200 psi

Figure 10(b).

A COMMERCIALY ATTRACTIVE THERMOPLASTIC ADHESIVE - ISOMER OF LARC-TPI

Donald J. Progar and J. Richard Pratt
Polymeric Materials Branch
Ext. 44256 February 1989
RTOP 506-43-11
Code RM WBS 52-2

Research Objective: This research was initiated to investigate the adhesive properties of a commercially attractive isomer of LARC-TPI, an LARC-developed polymer with exceptional adhesive and thermooxidative properties. Being very similar in chemical structure to LARC-TPI, the isomer should provide similar properties, and is potentially less expensive to produce. The new adhesive material is intended for use as a high temperature, high performance adhesive for future aerospace applications.

Approach: LARC-TPI is formed from the reaction of two monomers, an inexpensive dianhydride and an expensive diamine that is also a potential carcinogen. When cured, LARC-TPI is a versatile linear, thermoplastic polyimide polymer. The isomer of LARC-TPI was formed from an inexpensive diamine and a potentially inexpensive dianhydride. Molecular weight control of this isomer was achieved by endcapping. The glass transition temperatures (T_g) of the two materials were measured to determine if the chemical structure of the TPI-isomer provided a T_g close to that of LARC-TPI. The TPI-isomer was evaluated as an adhesive by preparing an adhesive tape and bonding titanium alloy (Ti-6Al-4V) adherends under a variety of conditions to optimize the bonding procedure. In order to compare the durability of the TPI-isomer with LARC-TPI, lap shear specimens were exposed to thermal aging and water-boil conditions.

Accomplishments: The new dianhydride monomer was synthesized in our laboratory and used with a commercially supplied diamine to prepare the TPI-isomer. As shown in the figures, LARC-TPI and the TPI-isomer have very similar structures. The T_g of the prepared polymer was determined to be the same as that of LARC-TPI, 260°C. An adhesive bonding procedure was developed which provided lap shear strengths comparable to those of LARC-TPI (see figure). Tests were performed at RT, 204 and 232°C. The lap shear strengths after thermal exposure at 204°C for 1000 hr were similar for both adhesive systems. In addition, the results of a 72-hr water-boil showed a moderate improvement in strength retention for the TPI-isomer over the LARC-TPI.

Significance: This new polymer shows the excellent adhesive and thermooxidative properties of LARC-TPI, but is prepared from monomers that eliminate the expense and potential carcinogenicity of the diamine used to prepare LARC-TPI. This molecular design technique of interchanging chemical units within a polymer structure also opens an entirely new area for future research.

Future Plans: Scale-up of the dianhydride monomer will be initiated so that a comprehensive study of the TPI-isomer can be completed. A more exhaustive adhesive study of the material is warranted because of the promising initial results. An evaluation of the polymer as a matrix resin for high temperature, high performance composites is a logical follow-on application for a commercially attractive high performance polymer.

Figure 11(a).

A COMMERCIALY ATTRACTIVE THERMOPLASTIC ADHESIVE

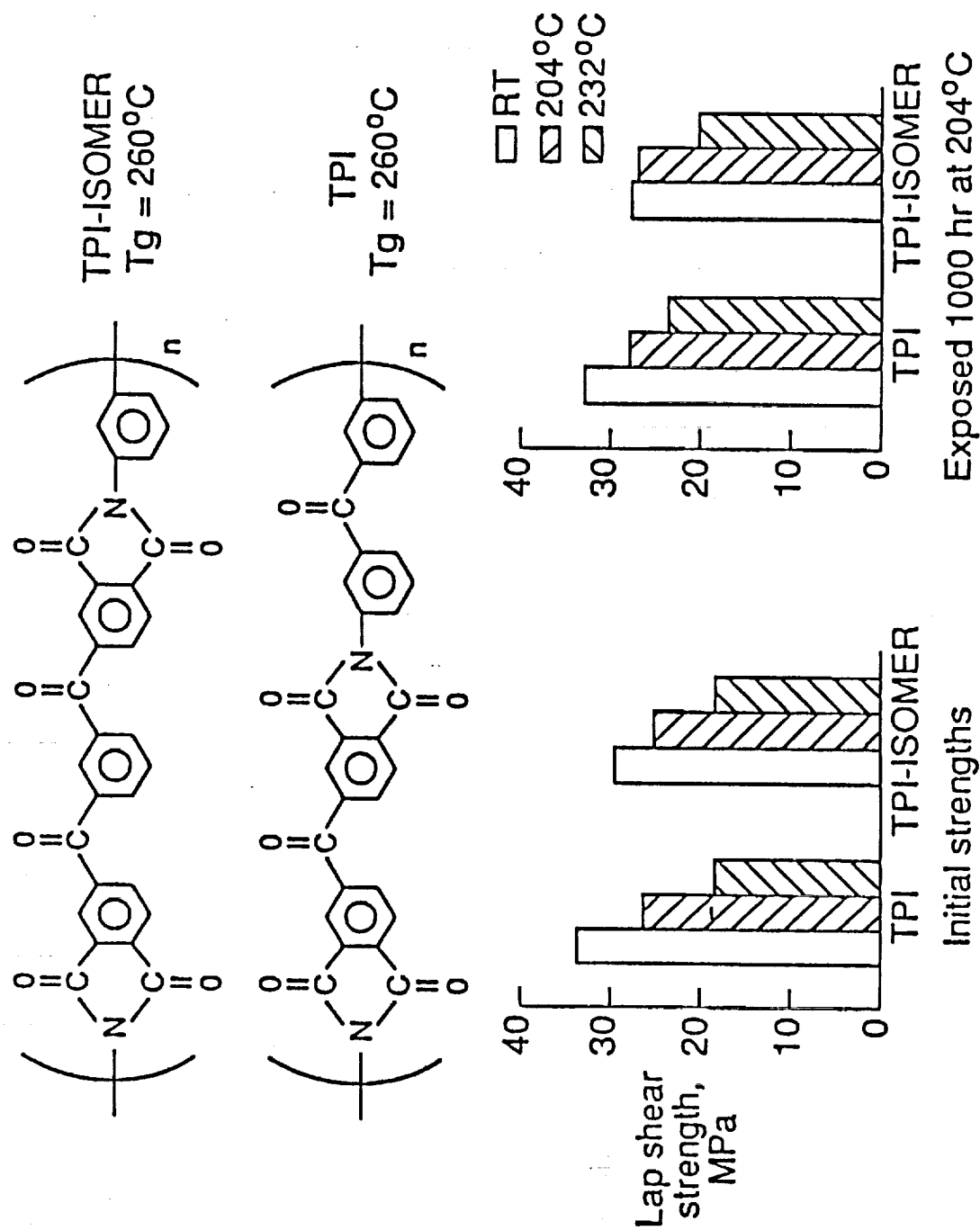
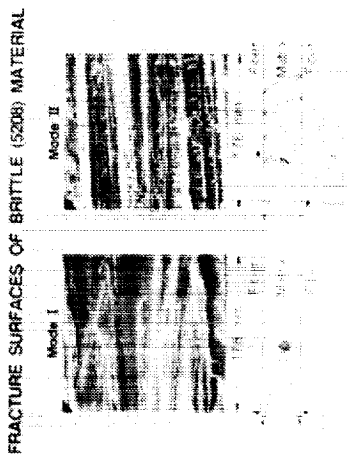


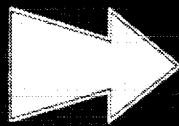
Figure 11(b).

MECHANICS OF MATERIALS BRANCH



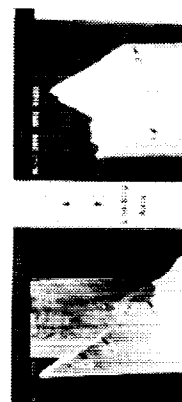
Polymeric Composites

- Experimental
- Theoretical Mechanics



- Constitutive Relationships
- Strength Models
- Fracture Mechanics
- Fatigue Life Models
- Microstructural Mechanics

ALUMINUM LITHIUM ALLOY 2090

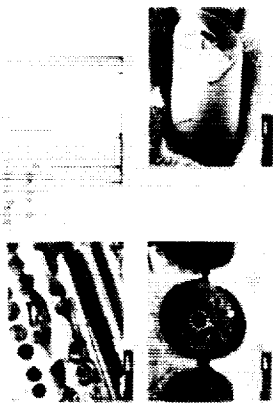


Light Metallic Alloys

- Prediction Methodology
- Improved Materials

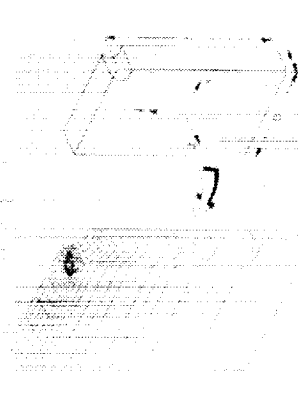


FIBER MATRIX FRACTURE



Metal Matrix Composites

SURFACE CRACK FROM A SEMI-CIRCULAR HOLE



Computational Mechanics

Figure 12.

MECHANICS OF MATERIALS BRANCH
FIVE YEAR PLAN

MAJOR THRUST	FY89	FY90	FY91	FY92	FY93	EXPECTED RESULTS
Airframe Materials (Base program)						Improved strength and life prediction methodologies
ACT						Improved strength and life prediction methodologies
HSR						Improved F/M Interface and optimum fibers
Generic Hypersonics						Improved F/M Interface and optimum fibers
Aging Aircraft						Improved F/M Interface and optimum fibers
Non-QASI						Applied fracture mechanics methodologies for special applications
ALS						Applied fracture mechanics methodologies for special applications
Fracture Control						Applied fracture mechanics methodologies for special applications
NASP						Applied fracture mechanics methodologies for special applications

Figure 13.

EFFECT OF FIBER/MATRIX INTERFACE STRENGTH ON THE MECHANICAL PROPERTIES
OF A SILICON CARBIDE/TITANIUM METAL MATRIX COMPOSITE

W. S. Johnson, R. A. Naik, and W. D. Pollock
Mechanics of Materials Branch
Ext. 43463 June 1989
RTOP 505-63-01
Code RM WBS 54-3

Research Objective: To determine the effect of a high temperature cycle, that is representative of a diffusion bonding process, on the mechanical properties of a titanium matrix composite.

Approach: The material system studied was a [0/90/0] layup of a silicon carbide fiber (SCS-6) reinforced titanium matrix (Ti-15-3) composite. One panel was subjected to 1000°C for one hour to simulate the temperature cycle that the material would see during a superplastic forming/diffusion bonding (SPF/DB) process. Another panel was left in the as-fabricated (ASF) condition. Both panels were cut into "dogbone" shaped specimens and tested at room temperature. Static tests were conducted to determine tensile strength and moduli. Several fatigue tests were conducted for each specimen type to create a cyclic stress range versus number of cycles to failure (S-N) curve. The Ti-15-3 matrix material was also tested in the ASF and SPF/DB conditions to assess changes in strength and moduli. Fibers were leached from both panels and tested for strength and modulus. The fracture surfaces were examined to assess changes in the failure modes.

Accomplishment Description: The composite showed a 9 percent increase in elastic Young's modulus, a 25 percent decrease in tensile strength and a 30 percent decrease in failure strain as a result of exposure to typical SPF/DB conditions. Furthermore, the fatigue endurance limit, shown in the figure, for the SPF/DB specimens was 50 percent lower than for the ASF specimens. The fracture surface of the ASF specimens, also shown in the figure, was very irregular with significant fiber pull-out as compared to the planar fracture surface of the SPF/DB specimen where fibers were flush with the surface. These features suggest that the fiber/matrix interface is stronger after the temperature cycle. A matrix crack growing towards a fiber with a stronger fiber/matrix interface is more likely to fracture the fiber rather than deflect along a weaker crack path such as an interface debond. This results in a more "notch-sensitive" material which will have a lower strength and fatigue life. Microstructural examinations of the fiber/matrix interphase regions also support the conclusion that the interface strength has increased. Additional tests showed that the SPF/DB matrix material had a higher modulus and strength than the ASF material. This would allow higher thermal residual stresses to be present in the SPF/DB composites which would also contribute to a higher interface bond strength. Therefore, the strength reduction exhibited by the composite is due to both a stronger fiber/matrix interface bond and an elevation of the residual radial compressive stresses around the fiber. The fiber properties were unaffected by the temperature cycle.

Significance:

This study shows that a typical SPF/DB thermal process cycle can result in a significant reduction in the tensile strength and the fatigue life of a composite despite the fact that both the matrix and fiber/matrix interface strengths are increased. In short, a stronger fiber/matrix interface may not always have a desirable effect on the performance of a composite. In general, a strong fiber/matrix interface is less desirable when designing a composite material system with a stronger and stiffer matrix.

Future Plans: The effects of different fiber coatings on composite mechanical properties will be studied. Compliant coatings such as gold and silver will be studied to assess their effects on residual stresses. Coatings such as TiB₂ will be assessed to determine differences in fiber/matrix reactions and strengths.

Figure 14(a).

EFFECT OF FIBER/MATRIX INTERFACE STRENGTH ON PROPERTIES OF TITANIUM MMC

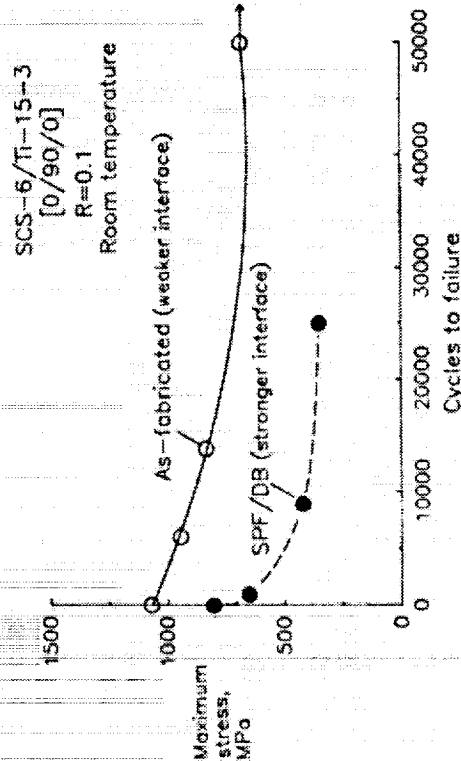
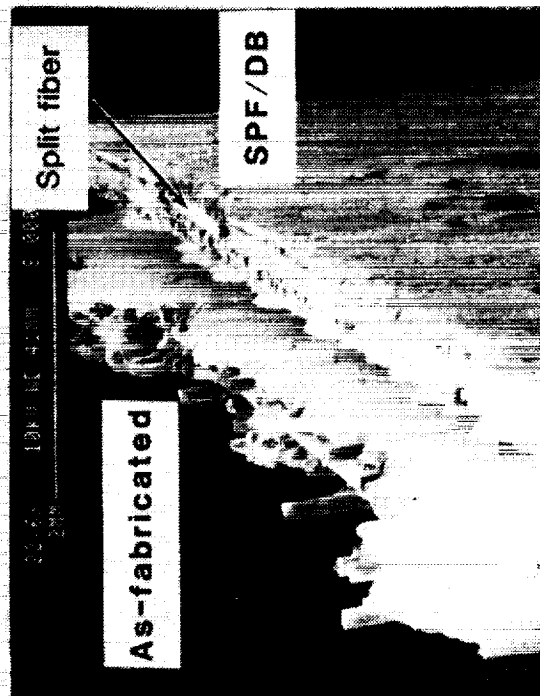


Figure 14(b)

MICROSTRUCTURE/MECHANICAL PROPERTY RELATIONSHIPS FOR DISCONTINUOUS REINFORCED METAL MATRIX COMPOSITES

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Mechanics of Materials Branch
Ext. 43463 September 1989
RTOP 506-43-71
Code RM WBS 54-3

Research Objective: To relate the microstructural characteristics of discontinuous reinforced metal matrix composites (MMC) to mechanical properties through the use of appropriate micromechanics models.

Approach: Powder metallurgy aluminum alloy 2124 in the unreinforced state and with discontinuous reinforcements of 15% and 30% SiC whiskers and 15% SiC particulates were manufactured in three plate thicknesses of 6.35, 3.18, and 2.8 mm and were investigated in the as-fabricated and peak-aged condition. Image analysis was used to assess the influence of the mechanical working on the reinforcement size, distribution, and orientation. Mechanical tests were then performed on each combination of reinforcement and material thickness to determine both longitudinal and transverse elastic moduli, Poisson's ratio, proportional limit (yield strength), and ultimate strength.

Accomplishment Description: The upper left figure shows a typical section of the 15% whisker reinforced MMC. The material is orthotropic on the microstructural scale. The upper right figure shows the average whisker (fiber) length for each plate thickness and the associated whisker orientations. The average whisker length decreases with decreasing plate thickness. This is because the whiskers are broken during the mechanical working required to reduce the plate thickness. The lower right figure shows how our micromechanics model introduces load into the whisker (fiber) by shear transfer from the matrix as a function of the whisker aspect ratio and relative fiber to matrix moduli. The plotted results illustrate the relative load carrying capacity of the whiskers as a function of their aspect ratio (L/d). The lower left figure shows the elastic modulus predictions based solely on the quantified microstructural details and the micromechanics model. The base matrix modulus is presented for comparison. The predictions are all within 5 percent. While the results are not shown, excellent predictions were also made for the 30% whisker reinforced material and the 15% particulate reinforced material.

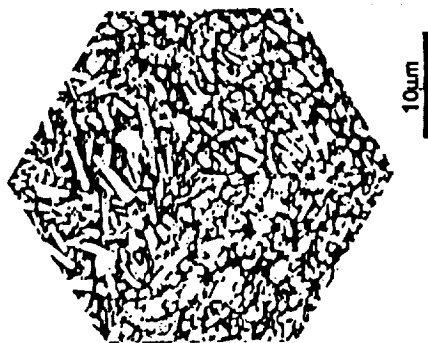
Significance: This study shows that a carefully conducted microstructural characterization of a discontinuously reinforced MMC can yield the required information to use with the appropriate micromechanics models to predict global composite properties such as moduli. Now that this is established, the micromechanics models can be used to engineer new microstructure for MMC's to produce the desired mechanical properties.

Future Plans: The materials described above are now being subjected to fatigue tests so that the microstructural features can be correlated to damage initiation and propagation.

Figure 15(a).

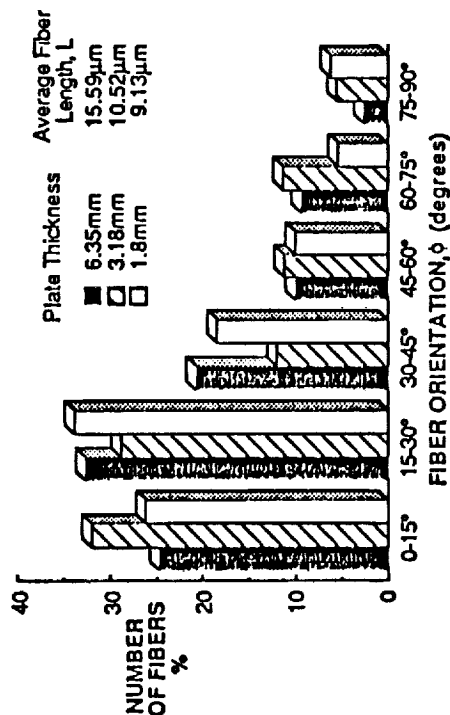
MICROSTRUCTURE / MECHANICAL PROPERTY RELATIONSHIPS FOR A DISCONTINUOUS ALUMINUM ALLOY MMC

MICROSTRUCTURE OF 2124 AL ALLOY
+ 15% SiC WHISKERS

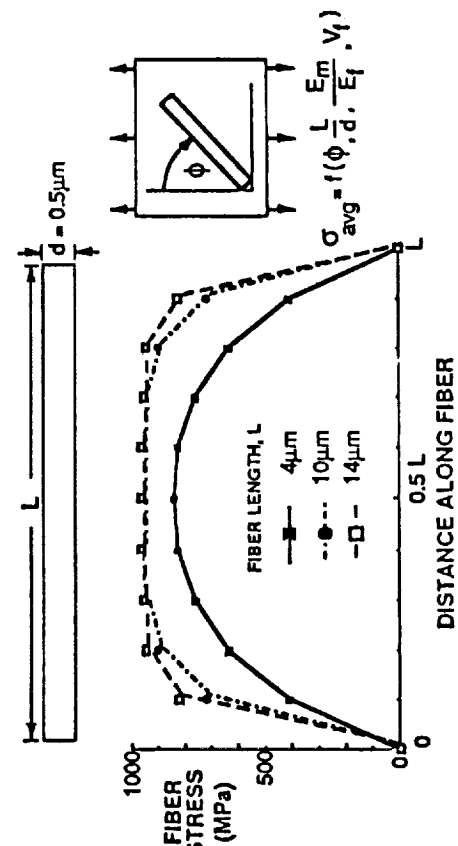


MMC IS VERY
ORTHOTROPIC
ON THE
MICROSTRUCTURAL
SCALE

ORIENTATION DISTRIBUTIONS



CALCULATED FIBER STRESS



YOUNG'S MODULUS

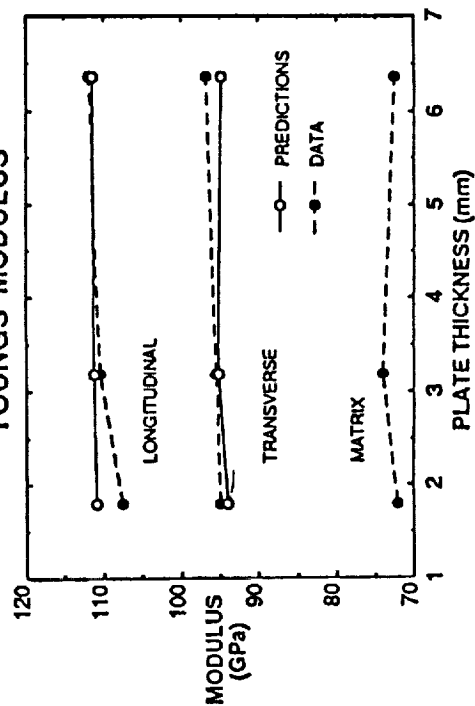


Figure 15(b).

C. C. Poe, Jr. and J. C. Newman, Jr.
 Ext. 43467 June 1989
 RTOP 506-43-11
 Code RM WBS 54-3

Research Objective: Light-weight pressurized fuel tanks used in launch systems and satellites are usually fabricated by welding. Because of the likelihood of weld defects, a tank must be proof tested to attain a high degree of reliability. For most metal alloys, crack-like flaws in welds can grow in size during a proof test, causing a loss in strength. Thus, a rational method is needed to ensure that operating pressures are low enough to allow for any damage caused by a proof test. Such a method is being developed using fracture mechanics.

Approach: The strength of 0.5-in.-thick 2024-T351, a "tough" aluminum alloy, is plotted against depth of a long surface crack in Fig. 16(b). "Lack-of-penetration" type defects in welds can behave like long surface cracks. The crack growth corresponding to a proof cycle and the subsequent operating cycle are shown for the case where the initial flaw depth is less by some arbitrarily small amount than that necessary to cause failure in the proof cycle. This initial crack depth gives the maximum stable crack growth and corresponding strength loss, which is about 8% in this case. The proof stress is assumed to be equal to the tensile yield strength. The operating stress shown in Fig. 16(b), which represents a typical proof factor (ratio of proof stress to maximum operating stress) of 1.05, is greater than the strength after the proof test. Thus, the pressure vessel would fail in operation if the initial crack depth were equal to that in Fig. 16(b). For more shallow cracks, the strength loss would be less. Thus, the proof factor must be greater than 1.08 unless some NDE method had been used after the proof test to screen out cracks as deep as that in Fig. 16(b). The crack growth and strength curves in Fig. 1 were predicted with a crack-growth resistance curve using fracture mechanics. This crack growth resistance curve is a material property like fracture toughness.

Accomplishment Description: Following the procedure outlined in Fig. 16(b), values of initial crack depth to cause failure in the proof test and values of proof factor to cause failure in operation were calculated for various values of proof stress and plotted in Fig. 16(c). For smaller initial crack depths and for larger proof factors, failure cannot occur in the proof test nor the subsequent operating cycle. Thus, the values of crack depth and proof factor in Fig. 16(c) are referred to as maximum and minimum values, respectively. The minimum proof factors increase with increasing proof stress and thickness, and the maximum initial crack depths decrease with increasing proof stress and increase with increasing thickness. Results are shown in Fig. 16(d) for 0.5-in.-thick 7075-T651 and 2024-T351. Although the 7075-T651 aluminum is less "tough" than the 2024-T351 aluminum, it exhibits similar stable crack growth. Thus, the curves of maximum initial crack depth are lower for 7075-T651 than for 2024-T351, but the curves for minimum proof factor cross and are not much different.

Significance: Operating stresses that will prevent failure from damage caused by a proof test depend on material, thickness, and proof stress. For 1/4-in. and thicker 2024-T351 and 7075-T651 aluminum plates, the corresponding proof factors are greater than the typical value of 1.05. Thus, current proof factors may be unconservative. Using resistance curves, minimum proof factors can be calculated for configurations other than long surface cuts, e.g. embedded cracks, cracks growing from holes containing loaded fasteners, etc.

Future Plans: Evaluate multiple proof cycles for improving reliability. Evaluate the benefits of proof testing for "aging aircraft." Improve accuracy by incorporating non-linear fracture mechanics. Conduct tests to measure the crack-growth-resistance curves for aluminum-lithium alloys so minimum proof factors can be predicted for those alloys.

Figure 16(a).

STRENGTH LOSS DUE TO PROOF CYCLE

0.5-IN.-THICK 2024-T351 WITH LONG SURFACE CRACK

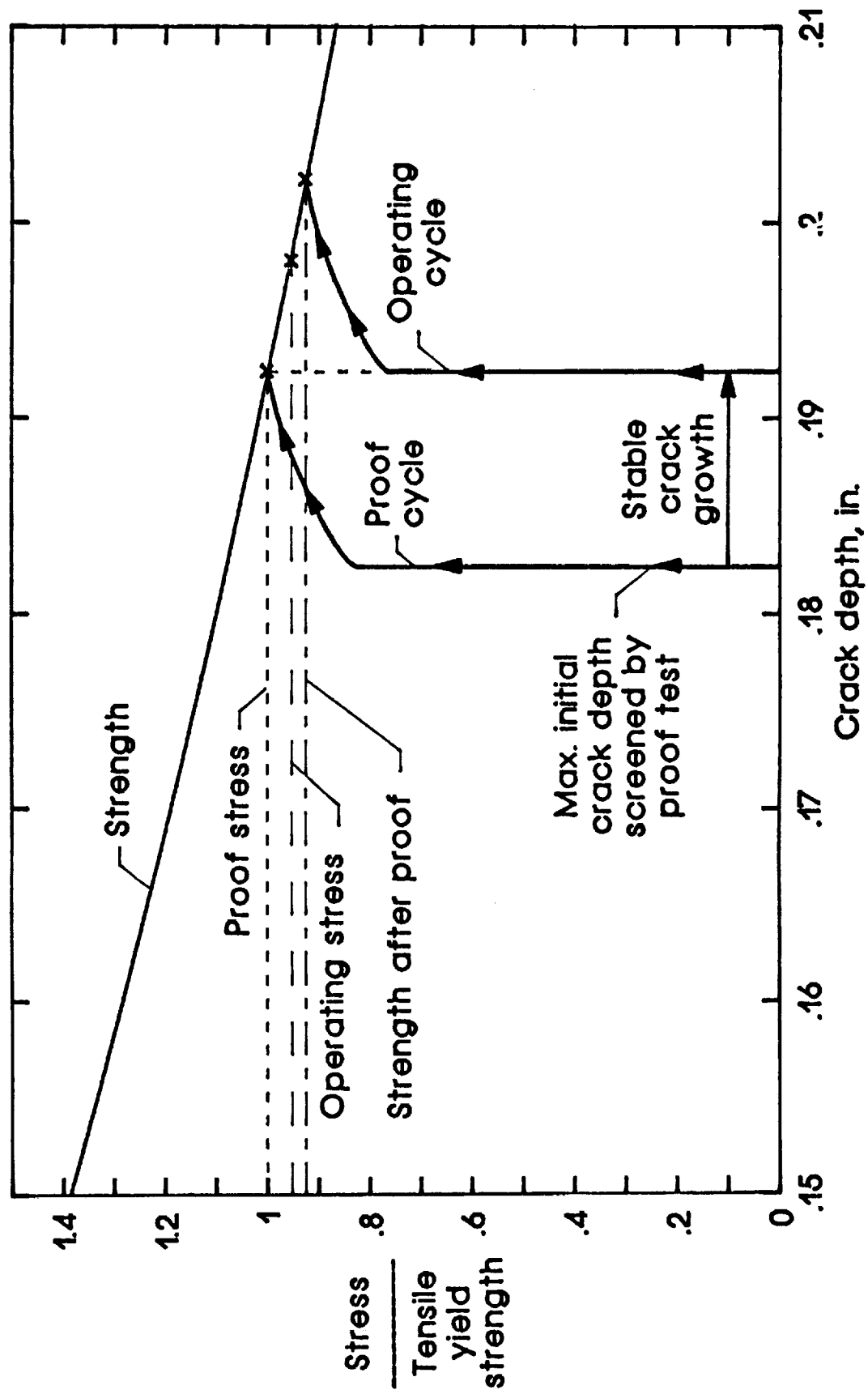


Figure 16(b).

MINIMUM PROOF FACTOR AND MAXIMUM INITIAL CRACK DEPTH

0.5-IN.-THICK ALUMINUM WITH LONG SURFACE CRACK

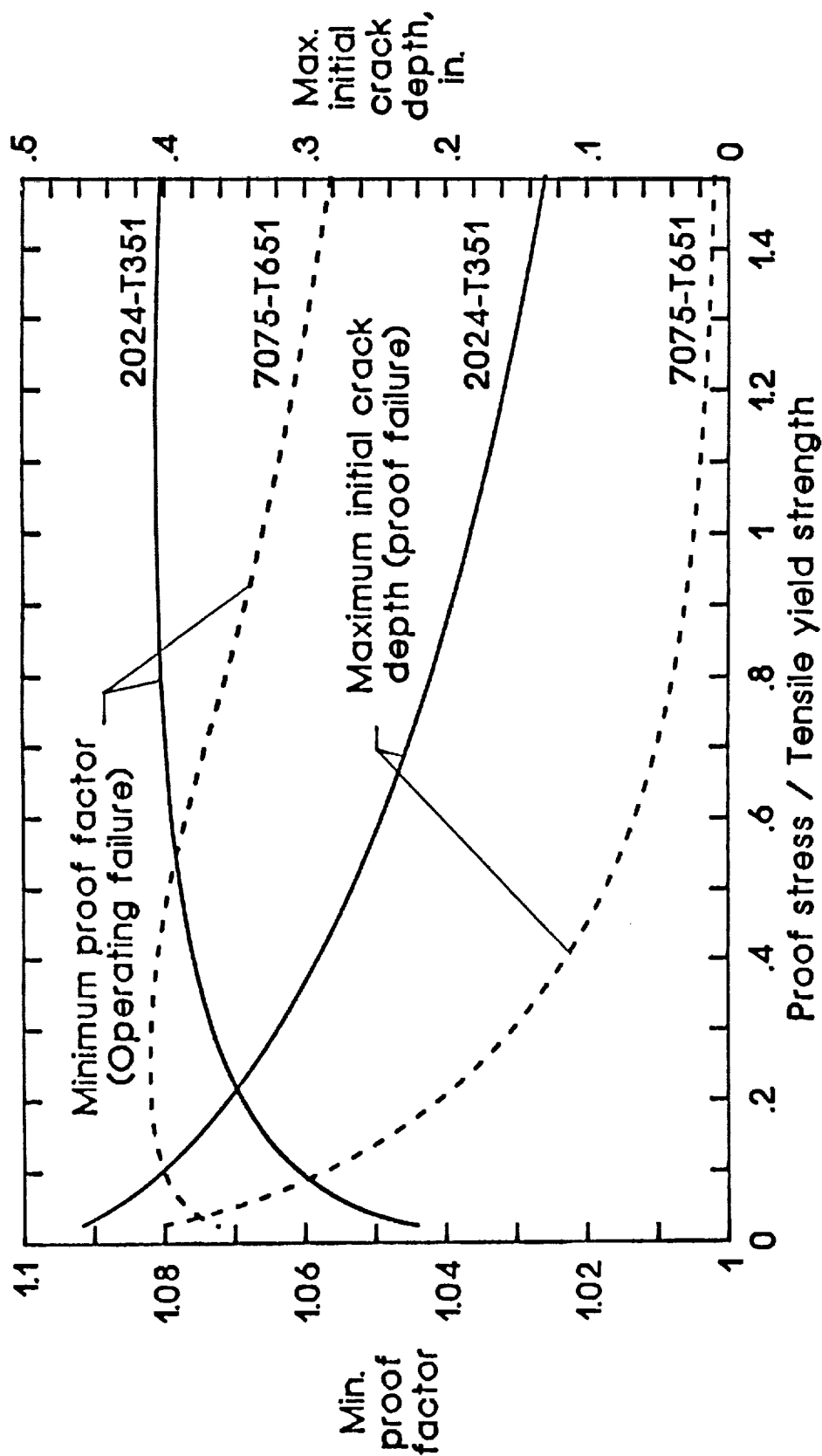


Figure 16(c).

MINIMUM PROOF FACTOR AND MAXIMUM INITIAL CRACK DEPTH

2024-T351 WITH LONG SURFACE CRACK

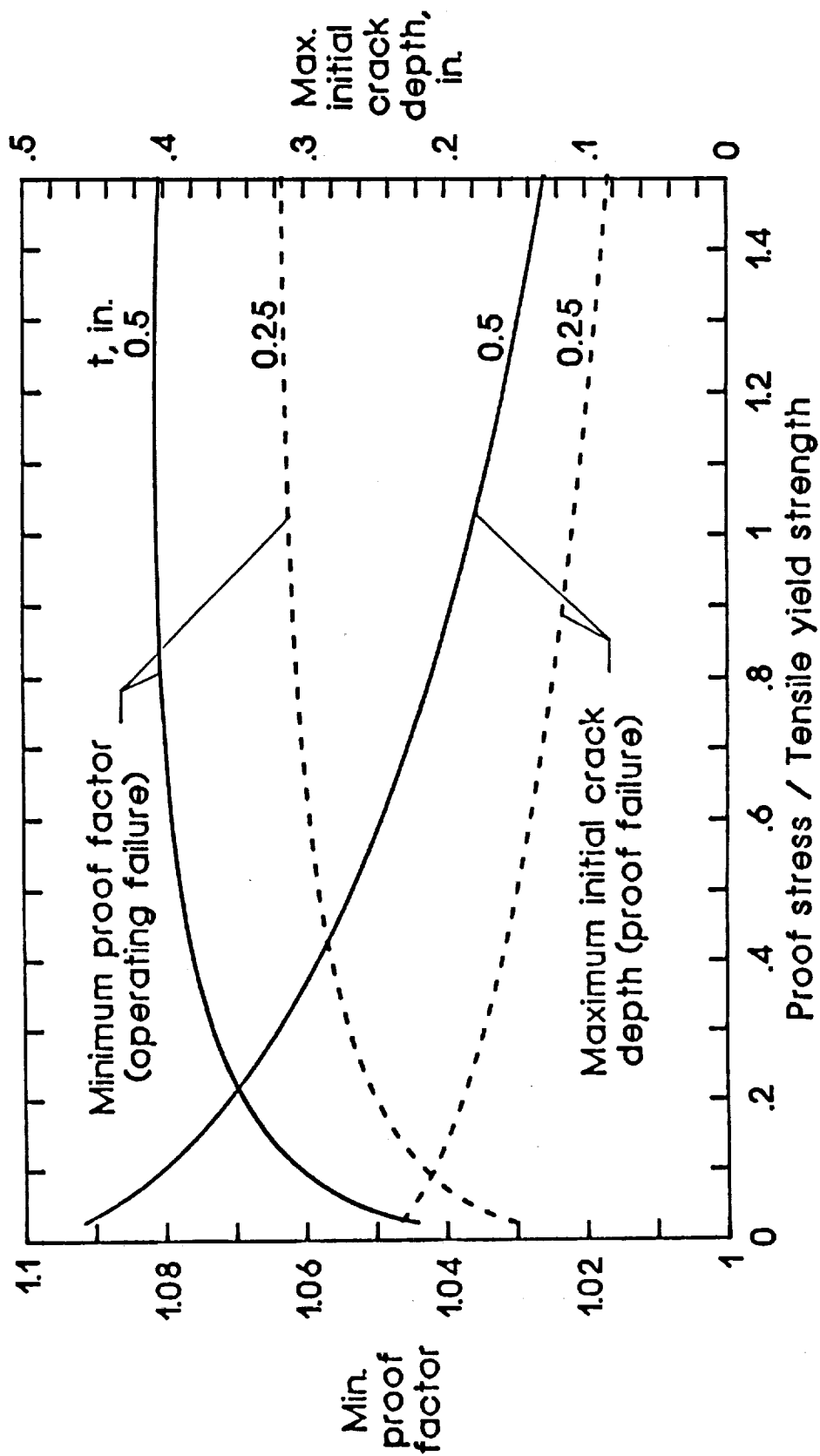


Figure 16(d).

A MIXED-MODE DELAMINATION TOUGHNESS TEST

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Mechanics of Materials Branch
Ext. 43456 October 1988
RTOP 505-63-01
Code RM WBS 54-3

Research Objective: To develop a test apparatus and procedure to measure mixed-mode delamination fracture toughness of composite materials.

Approach: Because composite structures can delaminate under complex loadings, composite materials should be tested with different combinations of mode I (opening) and mode II (sliding) loadings. A new mixed-mode bend (MMB) test was developed. The new test is a combination of a standard double cantilever beam (DCB) test for mode I and a standard end notch flexure (ENF) test for mode II. As shown in Fig. 17(b), this combination uses a lever to apply simultaneous DCB and ENF type loadings. By varying the position of the load on the lever, the mixture of mode I and mode II loading can be changed. Varying the loading mixture changes the relative amounts of mode I and mode II strain energy release rate (G_I and G_{II} , respectively) available to extend the delamination.

Accomplishment Description: A closed form equation as well as a finite-element analysis were developed to compute G_I/G_{II} ratios for the MMB test. Mixed-mode delamination toughness tests were performed in a displacement control test machine to measure the mixed-mode delamination toughness of a tough graphite/thermoplastic composite, AS4/PEEK (APC2). Three mixed-mode ratios were used along with pure mode I and pure mode II cases. The data from this study are shown in Fig. 17(c) as the mode I and mode II components of mixed-mode fracture toughness (G_{IC}^m and G_{IIc}^m , respectively). The curve is nearly horizontal in the region where $G_I/G_{II} > 1$, indicating that the toughness is nearly independent of G_{II} and, therefore, controlled by the mode I loading. In the region where $G_I/G_{II} < 1$, the curve is sloped indicating that both the mode I and mode II loadings influence the delamination toughness.

Significance: The MMB test solves many of the problems that exist with other types of mixed-mode delamination testing and uses a single specimen configuration to test over a wide range of mixed-mode G_I/G_{II} ratios. Also, a simple closed form equation has been developed to partition the measured mixed-mode toughness into its mode I and mode II components. The MMB test evaluates composites under mixed-mode conditions similar to those in aerospace structures and, therefore, should lead to an improved understanding of delamination and better predictions for delamination in composite structures.

Future Plans: The MMB test will be used to study the micromechanics governing mixed-mode delamination toughness. Also, cyclic MMB testing will be explored.



Figure 17(a).

THE MIXED - MODE BENDING TEST

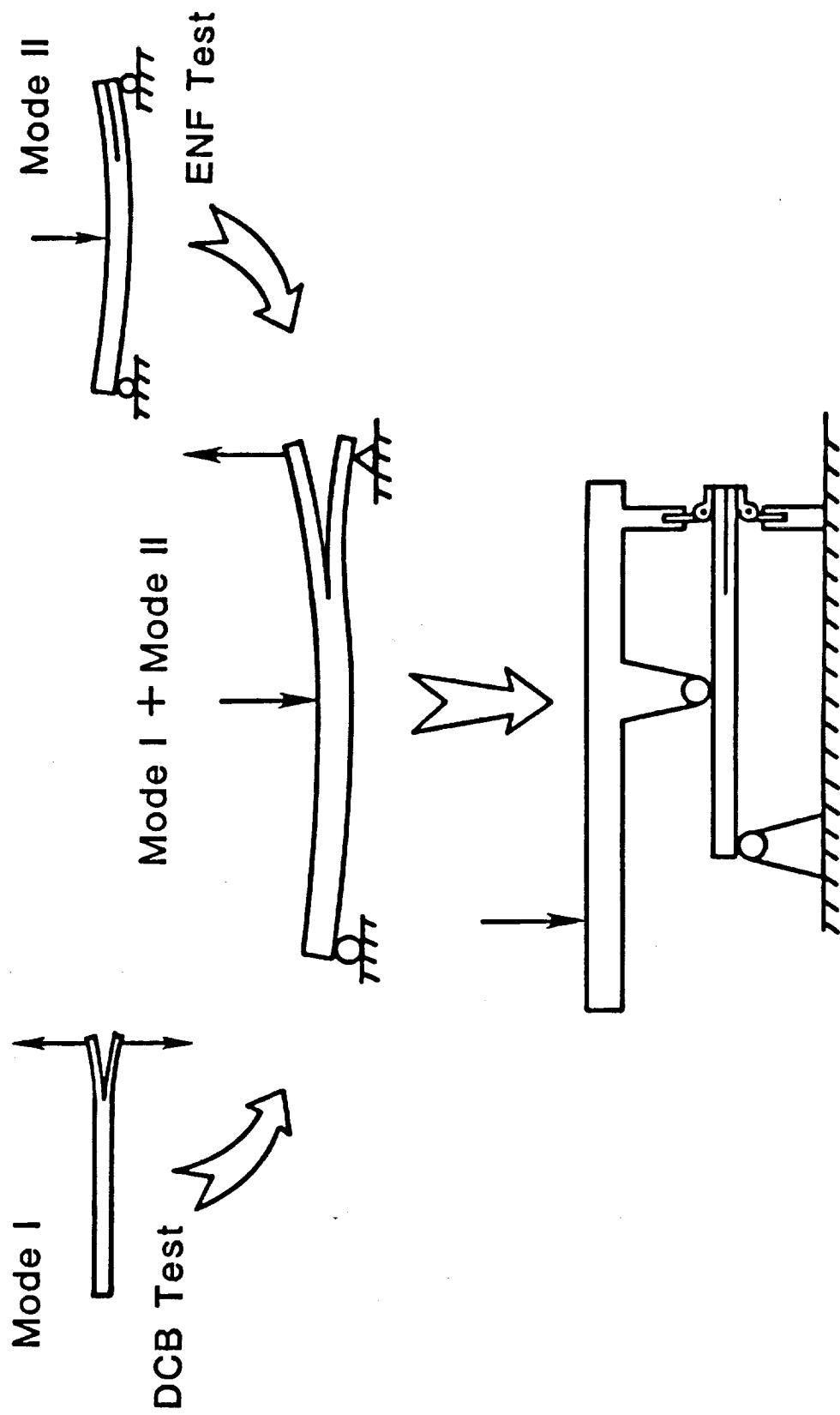


Figure 17(b).

MIXED-MODE DELAMINATION TOUGHNESS

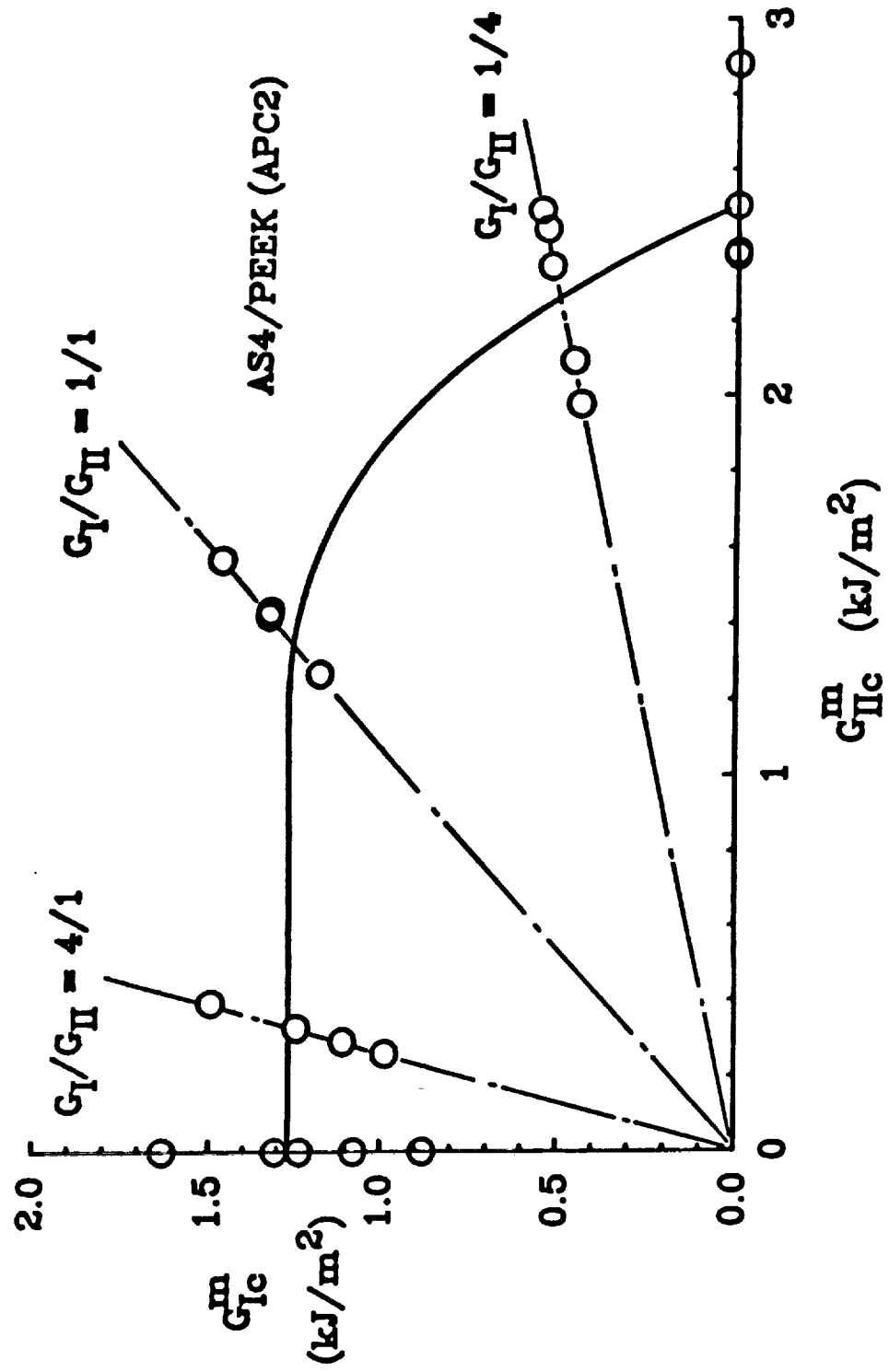


Figure 17(c).

FATIGUE LIFE OF STITCHED GR/EP LAMINATES

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Mechanics of Materials Branch

H. Benson Dexter and Marvin B. Dow
Applied Materials Branch

Ext. 43467 December 1988

RTOP 505-63-01

Code RM WBS 54-3

Research Objective: Stitching has been shown to improve the compression strength of graphite/epoxy laminates containing damage from low-velocity impacts. The objective of this investigation was to determine whether or not the stitching process itself damages the material and reduces fatigue life.

Approach: The plates were made by first stacking dry layers of "unidirectional" AS4 graphite fabric, stitching the stack of layers, and then introducing 3501-6 epoxy resin. The stack of layers was stitched in orthogonal directions with 1500 denier Kevlar thread 8 times per inch. The rows of stitching were spaced 1/4-inch apart (See figure). The plates contained a total of 40 layers oriented at 0°, 45°, -45°, and 90°, so that the plates were transversely or "quasi" isotropic. One-inch-wide fatigue specimens were cut from the stitched plates and tested in fatigue to failure. Holes of 1/4-in. diameter were made in the center of some of the specimens. Unstitched specimens were also tested for comparison. The cyclic fatigue loading was in compression (See figure).

Accomplishment Description: The maximum compression stress is plotted against the logarithm of cycles to failure in the figure. Regression lines are also plotted. Static compression strengths are plotted at 1 cycle. The fatigue strengths of stitched specimens without holes were less than those of unstitched specimens. But the fatigue strengths were essentially equal for stitched and unstitched specimens with holes, and considerably less than those of specimens without holes. Thus, while stitching reduces the compression fatigue strength of unnotched specimens, it has little effect on fatigue strengths of specimens with holes. It is conceivable that stitching could even increase static and fatigue strength for large holes or cutouts. Delaminations initiated at the edges of a specimen without holes just below the surface and propagated to the center, rendering the surface layers ineffective in compression. Then, another delamination initiated at the edges below the first delamination and propagated to the center, rendering more layers ineffective. This process repeated until finally the net compression stress exceeded a critical level and the specimen failed transversely by microbuckling. In specimens with holes, the delaminations initiated at the edge of the hole rather than at the edge of the specimen.

Significance: For graphite/epoxy laminates with impact damage, stitching can improve residual compression strengths significantly but not without some loss in unnotched fatigue strength or initial static strength. On the other hand, stitching may not reduce the fatigue strength or initial static strength of an actual structure where discontinuities like holes are normally present.

Future Plans: Additional experiments will be conducted to determine the effect of hot/wet conditions, impact damage, and loading spectra.

Figure 18(a).

COMPRESSION FATIGUE OF STITCHED AS4/3501-6

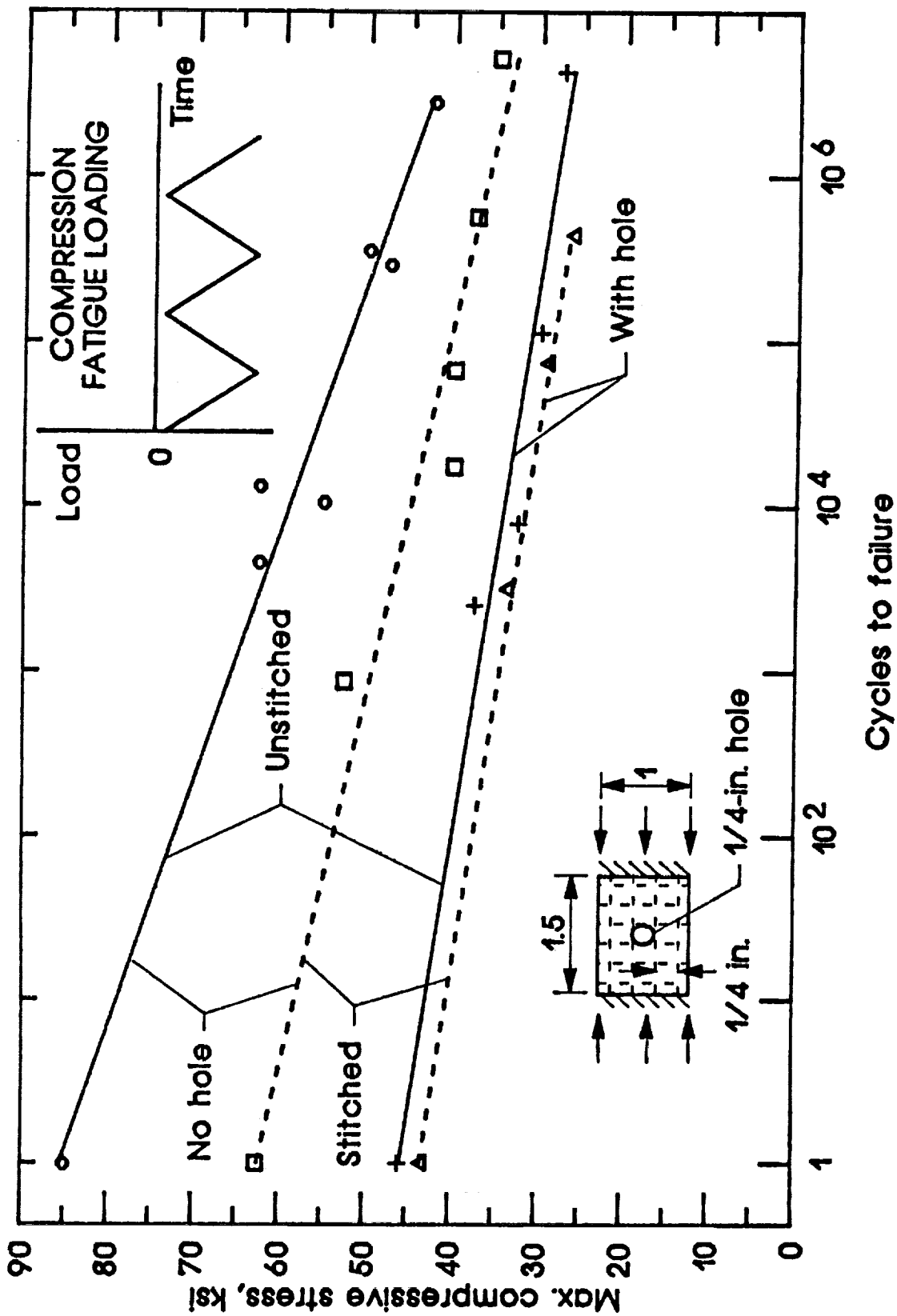


Figure 18(b).

A MICROMECHANICS TREATMENT OF THE EFFECTS OF CURVATURE
IN THE MAIN LOAD-CARRYING LAYERS ON COMPOSITE STIFFNESS

Charles E. Harris
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Ext. 43449 December 1988
RTOP 505-63-01
Code RM WBS 54-3

Research Objective: Develop a kinematically based micromechanics model to predict the effect of planned or manufacturing-induced curvature (waviness) in the main load-carrying layers on the stiffness of composites.

Approach: A simple mathematical model describing the deformation behavior of the main load-carrying layers of a laminated composite has been developed. The main load-carrying layers have initial curvature due to the manufacturing process and are modeled as curved beams supported by a continuous elastic foundation. The stiffness of the elastic foundation represents the constraint on the main load-carrying layer provided by the filler or matrix material. Model formulations include the geometry where the layers are exactly in-phase or parallel and where the layers are out-of-phase. The deformation behavior of the filler material for these two extreme cases is different and, therefore, necessitate a different formulation of the elastic foundation "spring" constants. The principle of minimum potential energy was used to develop the governing equations and the accuracy of the analytical results were examined by a carefully planned experimental program. Finally, the usefulness of the mathematical models was examined by several applications to "real" composites with in-situ local curvature.

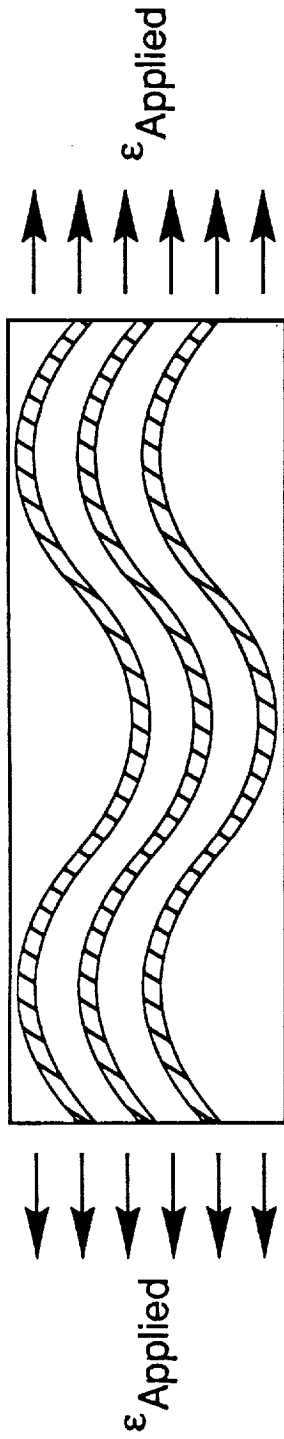
Accomplishment Description: The accuracy of the micromechanics model was certified by comparison to experimental results obtained from specially prepared specimens. Corrugated aluminum layers with precisely controlled curvature were bonded between layers of a room-temperature curing epoxy to fabricate specimens with both in-phase and out-of-phase wavy layer patterns. The effective Young's modulus (composite stiffness) predicted by the micromechanics model is compared to experimental results for a variety of geometries and specimen configurations in Figure 19(b). The mathematical expression for the effective Young's modulus of the in-phase pattern is given in the figure. As can be seen, the specimens with the in-phase pattern exhibited a more significant curvature effect than did the specimens with the out-of-phase wavy pattern. This is because the epoxy filler in the in-phase case, which is shear dominated, deformed more readily than the filler in the out-of-phase case which is dominated by tension-compression extensional behavior. Therefore, the epoxy filler provides less constraint against straightening of the main load-carrying layers in the in-phase case than in the out-of-phase case.

Significance: This simplified micromechanics model shows promise as a useful tool to evaluate the trade-off of in-plane properties of "three-dimensional" composites relative to "straight" tape layup laminates. The model may aid in tailoring advanced composite forms with planned fiber waviness.

Future Plans: The model will be used to develop a composite strength model based on the maximum strain in the main load-carrying layer. The model will also be extended to include the fiber-matrix interface shear transfer mechanisms so that realistic filler strains can be predicted.

Figure 19(a).

Micromechanics model of in-phase wavy layers



$$\eta = \frac{E_{WAVY}}{E_{STR}} = \frac{\left[\frac{2h_2^2 G}{h_2 - h_1} + \frac{2\pi^2 E h_1^2}{3L^2} \right]^2}{\frac{\pi^2 H_0 h_1 E}{2L^2} \left[\frac{4h_2^2 G}{h_2 - h_1} + \frac{4\pi^2 E h_1^2}{3L^2} \right]}$$

$$\frac{E_{WAVY}}{E_{STR}}$$

- E_{WAVY} = Effective laminate modulus with wavy layers
- E_{STR} = Effective laminate modulus with straight layers
- h_1 = half thickness of wavy layer
- h_2 = half thickness of filler and wavy layer
- L = pitch of initial wavy pattern
- H_0 = maximum height of initial wavy pattern
- G = shear modulus of filler
- E = Young's modulus of wavy layer

Figure 19(b).

Comparison of the effective laminate modulus due to initial curvature (waviness) of the main load-carrying layers ($H_0 = 0.078"$, $L = 0.51"$)

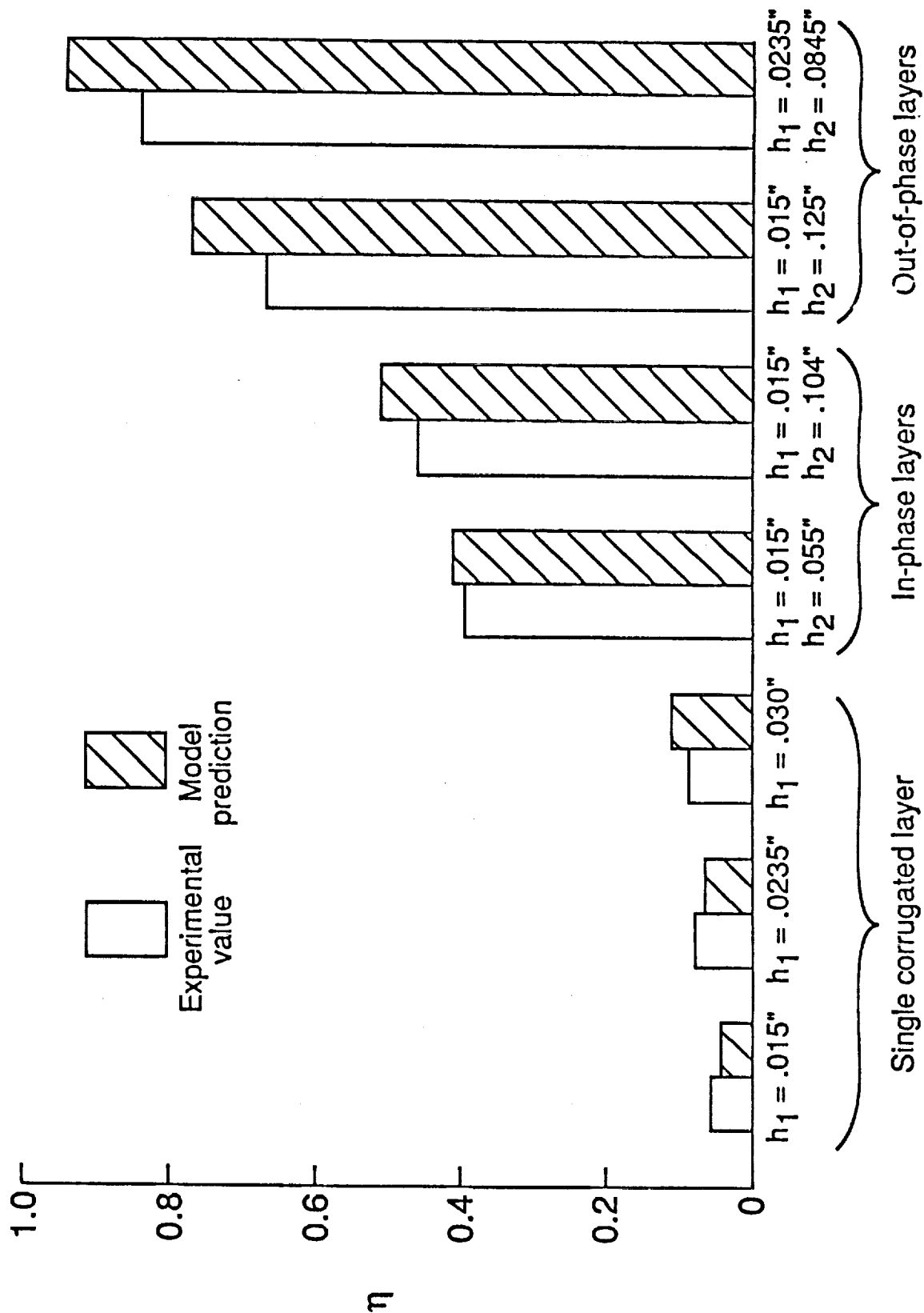


Figure 19(c).

ANALYSIS PREDICTS ONSET OF INSTABILITY-RELATED DELAMINATION GROWTH

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Ext. 43458 February 1989
RTOP 505-63-01
Code RM WBS 54-3

Research Objective: Develop analysis for predicting onset of Instability-Related Delamination Growth (IRDG).

Approach: Compression loads can cause a locally delaminated region to buckle, which sometimes leads to IRDG. Figure 20(b) shows a typical laminate with a buckled delaminated region. Based on previous work, it was expected that delamination onset could be predicted using fracture mechanics, in particular, strain-energy release rates. A nonlinear three-dimensional analysis NONLIN3D was developed which could efficiently determine the distributions of the components of strain-energy release rate (G_I , G_{II} , and G_{III}) along the delamination front. Six test specimen configurations were designed which were expected (based on the analysis) to exhibit significantly different behaviors. The material was IM7/8551-7 graphite epoxy. These specimens were fabricated with circular Kapton inserts to simulate an initial delamination. A comparison of experimental and analytical results was used to validate the analysis and to identify what governs the onset of IRDG.

Accomplishment Description: The graph in Figure 20(b) shows typical distributions of G_I and G_{II} along the delamination front. (The G_{III} is not shown since it was always negligible.) The analytical results correctly predicted the behavior of the experimental specimens, which exhibited delamination growth transverse to the load direction and only on a small portion of the delamination front. Figure 20(c) shows the maximum G_I and G_{II} along the delamination front for each configuration at delamination onset. Also shown is the range of critical mode I strain-energy release rate, G_{IC} , determined from double cantilever beam tests and reported by the materials manufacturer. These results indicate that delamination onset is predictable based on just G_I . The failure of G_I to predict the behavior of configuration 1 was due to the presence of failure modes (not included in the analysis) which apparently increased the interlaminar stresses.

Significance: These results show that the onset of instability-related delamination growth can be predicted using NONLIN3D. The ability to predict growth will add greater precision to flaw criticality assessments as part of a damage tolerance methodology.

Future Plans: No new research in this area is planned. The methodology and results will be disseminated to industry. The analysis NONLIN3D will be evaluated for submission to COSMIC.

Figure 20(a).

Laminate With Postbuckled Delaminated Region

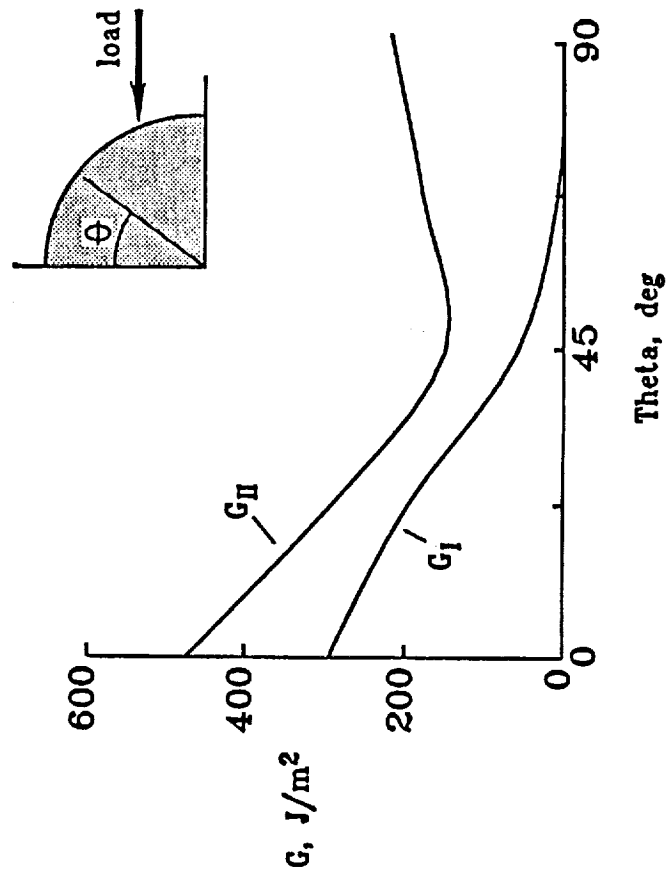
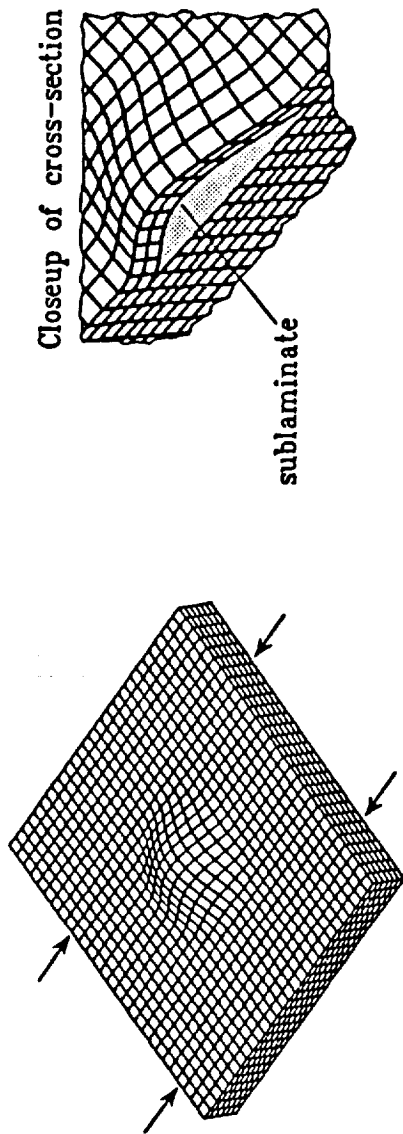


Figure 20(b).

Strain-Energy Release Rates for Delamination Initiation

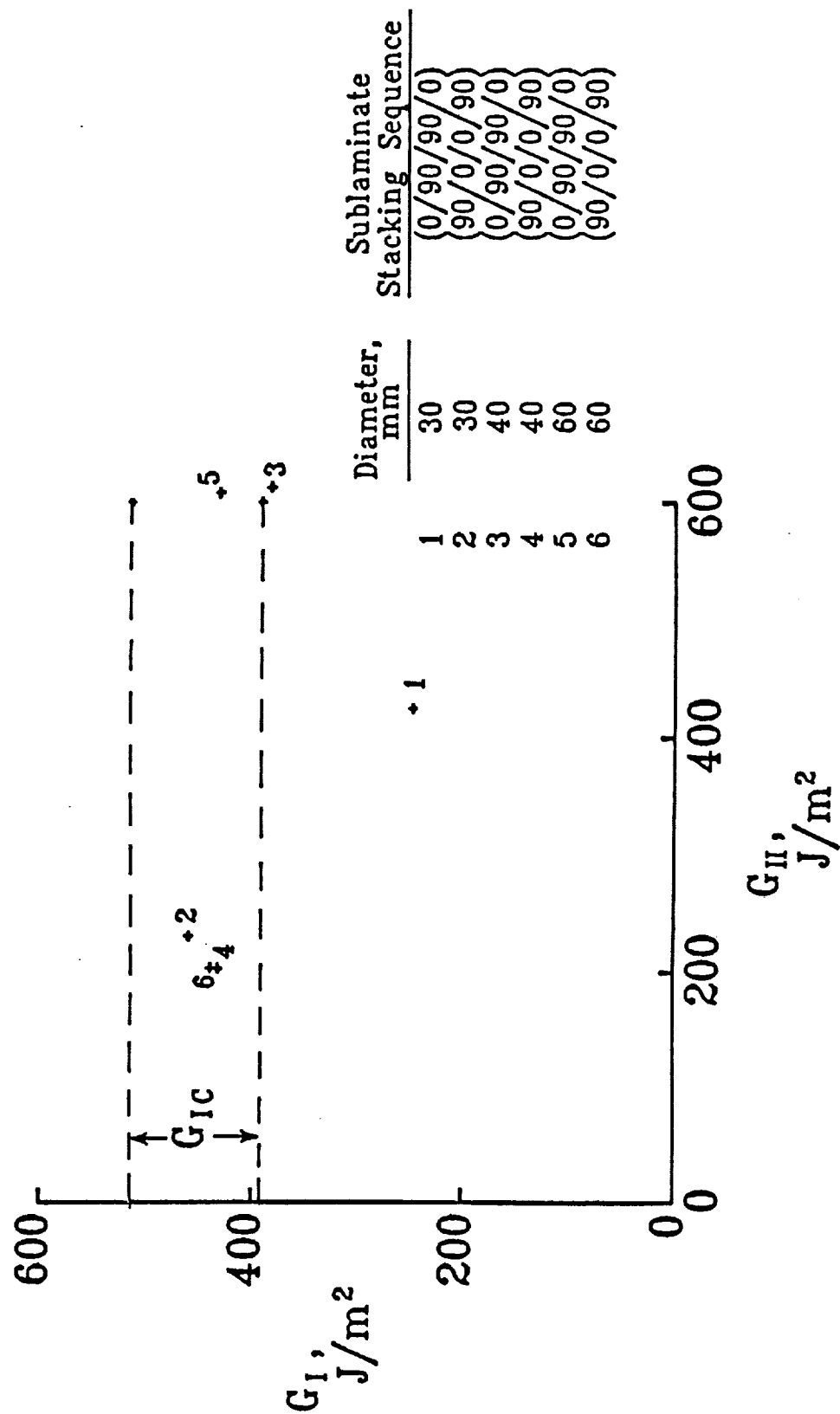


Figure 20(c).

STRAIN ENERGY RELEASE RATE ANALYSIS OF DELAMINATION IN A TAPERED
LAMINATE SUBJECTED TO TENSION LOAD

T. K. O'Brien, S. A. Salpekar, and I. S. Raju
Mechanics of Materials Branch
Ext. 43465 April 1989
RTOP 505-63-01
Code RM WBS 54-3

Research Objective: Determine the strain-energy-release rate for delamination growth in a composite tapered beam with internal plydrops.

Approach: Tapered composite laminates are used in rotor hubs and in a variety of other composite structures to tailor thickness. The laminate cross section is reduced by terminating some plies internally along the length of the beam. Such laminates have a tendency to delaminate at the interface where plies are terminated. The location for delamination onset was determined from the interlaminar normal stress distribution at the interface. Finite-element analyses were performed to calculate the total strain-energy-release rate, G , and its components, associated with the delamination. The stability and the direction of the delamination growth were also investigated.

Accomplishment Description: Steep gradients in the interlaminar normal stress distributions occurred at the transition of the tapered and the thin regions of the laminate (point C in Figure 21(b)). Therefore, this location was assumed to be the site for delamination onset. A certain delamination length was assumed along the taper and the G values were calculated for delamination growth in the thin region. The peak value of G in this distribution was considered to initiate delamination growth. Similarly, the peak G was obtained for delamination growth along the taper by assuming a fixed delamination length in the thin region. The peak G values both along the taper, and in the thin region, increase with longer delamination on the other side and have comparable values (Figure 21(c)). Hence, once a delamination forms on either side of point C, it will also grow on the other side as well, resulting in an unstable delamination growth.

Significance: This study indicates that a delamination initiating at the location of peak interlaminar normal stress in a tapered laminate will grow unstably and simultaneously in both directions.

Future Plans: The finite-element results will be compared with strain-energy-release rate predictions from a simple fracture mechanics model.

Figure 21(a).

TAPERED LAMINATE CONFIGURATION AND LOADING

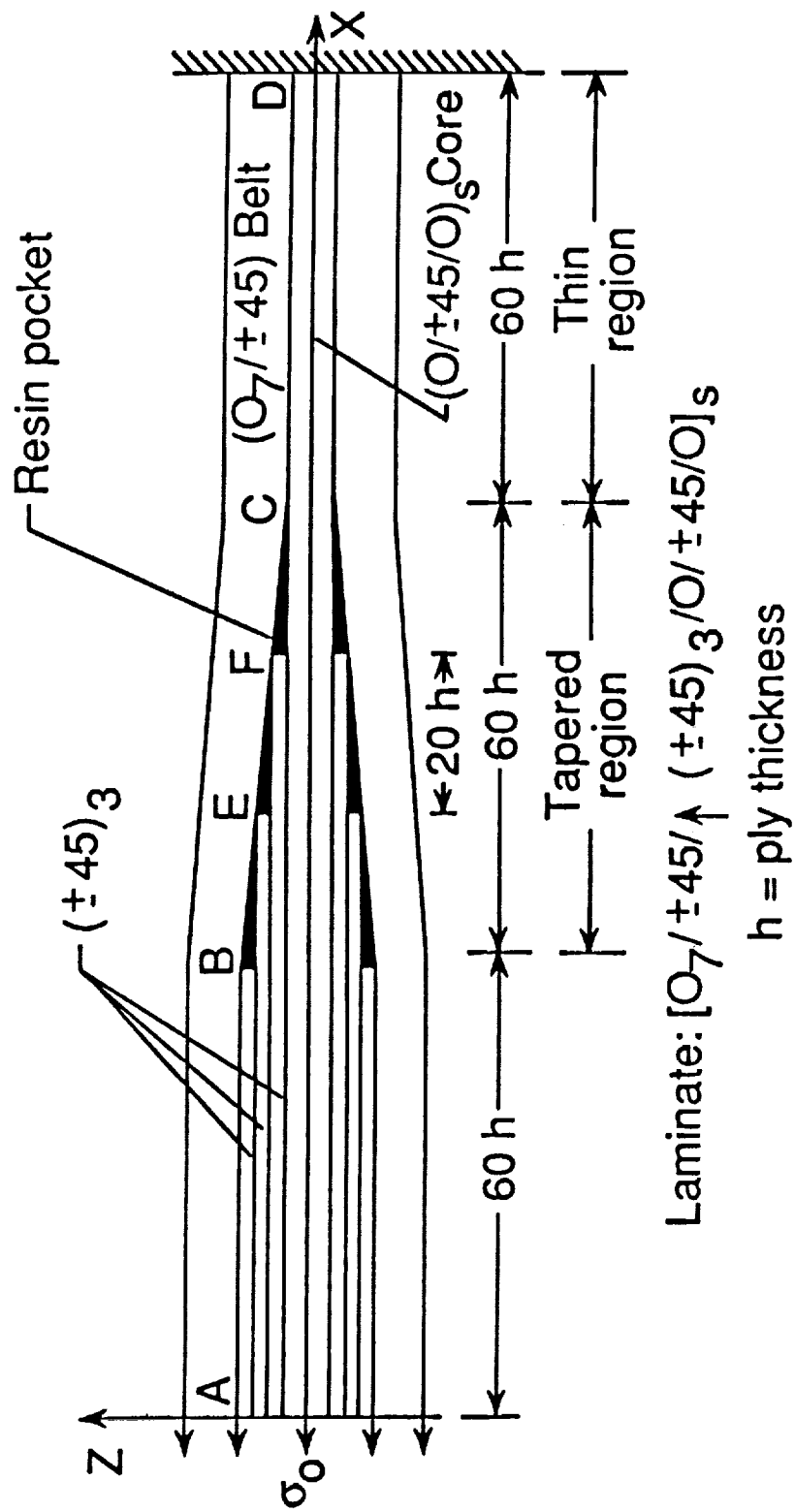


Figure 21(b).

PEAK VALUES OF TOTAL STRAIN ENERGY RELEASE RATE

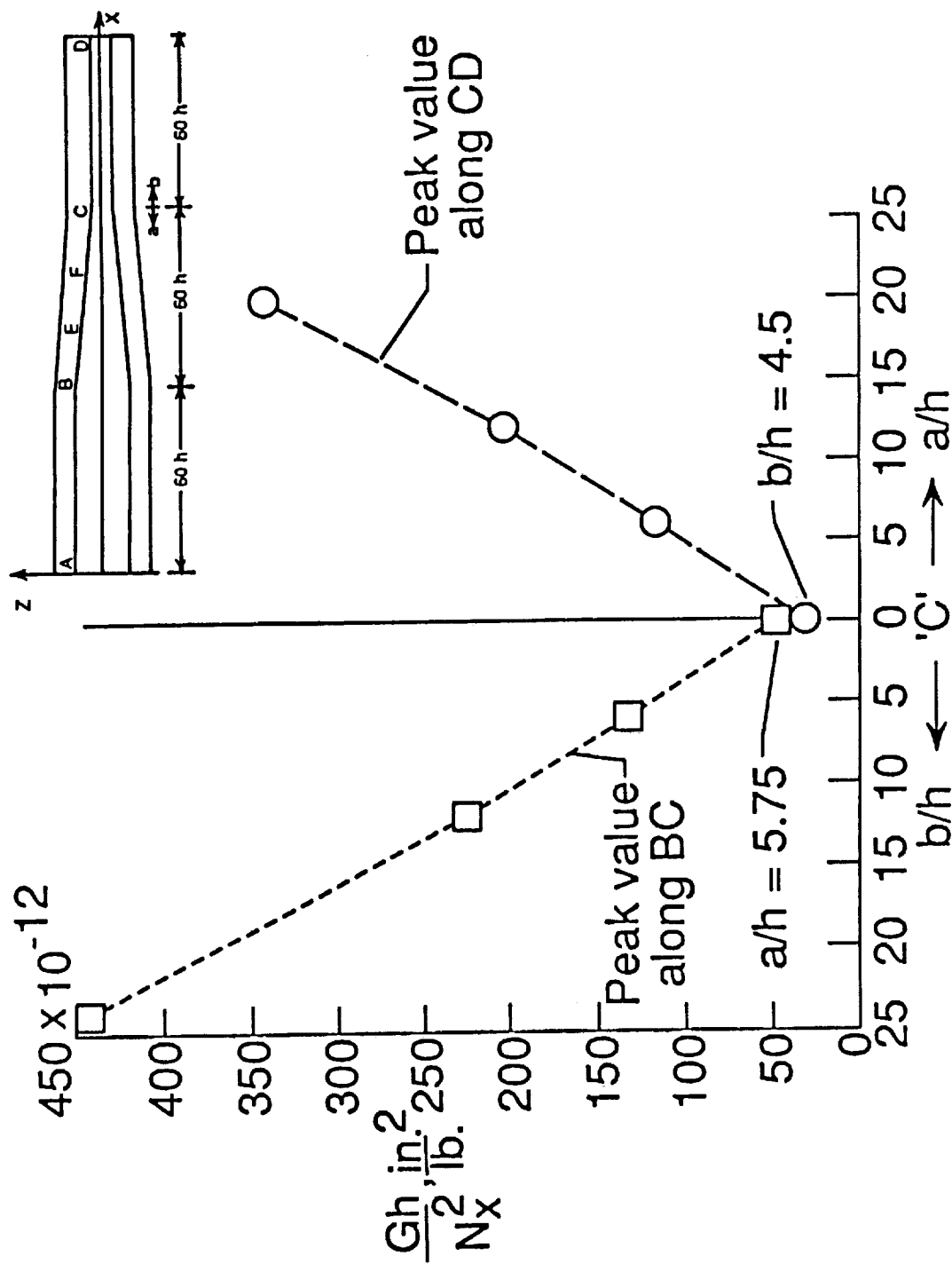


Figure 21(c).

WATER INTRUSION IN THIN-SKINNED COMPOSITE
HONEYCOMB SANDWICH STRUCTURES

Wade C. Jackson and T. Kevin O'Brien
Mechanics of Materials Branch
Ext. 43468 April 1989
RTOP 505-63-01
Code RM WBS 54-3

Research Objective: Structural components consisting of a honeycomb sandwiched between two thin composite skins are highly desirable for many aircraft applications. However, a pressure difference can drive water into damaged components causing an increase in weight, damage to cell walls, and debonds between the skin and honeycomb. The purpose of this study was to determine the effects of impact damage and cyclic loads on the susceptibility to water intrusion in thin-skinned composite honeycomb sandwich structure.

Approach: An exploratory investigation was conducted on thin-skinned composite honeycomb sandwich structure taken from the rotor blades of the Boeing Chinook and McDonnell Douglas Apache helicopters. An apparatus was developed to apply a vacuum on one side of the glass/epoxy skin so that a corresponding flow could be measured on the opposite side. Minimum impact and fatigue conditions were determined which would create microcracks large enough for water to flow through. Flow rate variations with applied strain and pressure difference were also investigated.

Accomplishment Description: The flow rate of air and water through a skin was found to be a function of moisture content, damage, applied strain, and pressure difference. These flow rates tended to increase exponentially as the number of load cycles, applied strain, or the pressure difference increased. A skin must have damage present for air and water to pass through. The air intrusion test was a good indicator of damage in the form of matrix cracks and has possible applications as a laboratory NDI method for thin composite laminates. The strain amplitudes necessary to grow microcracks within one million load cycles in the Chinook skin were found to be well above the levels recorded during flight certification (see figure).

Significance: If water is intruding through the rotor blade skin early in the blade service life, the water may be coming through damage resulting from an impact or damage developed under high cyclic loads in excess of those observed during flight certification tests.

Future Work: No future work is currently planned.

Figure 22(a).

THRESHOLD ϵ_{\max} FOR INCREASED FLOW RATE AFTER CYCLING AT $R = 0.1$

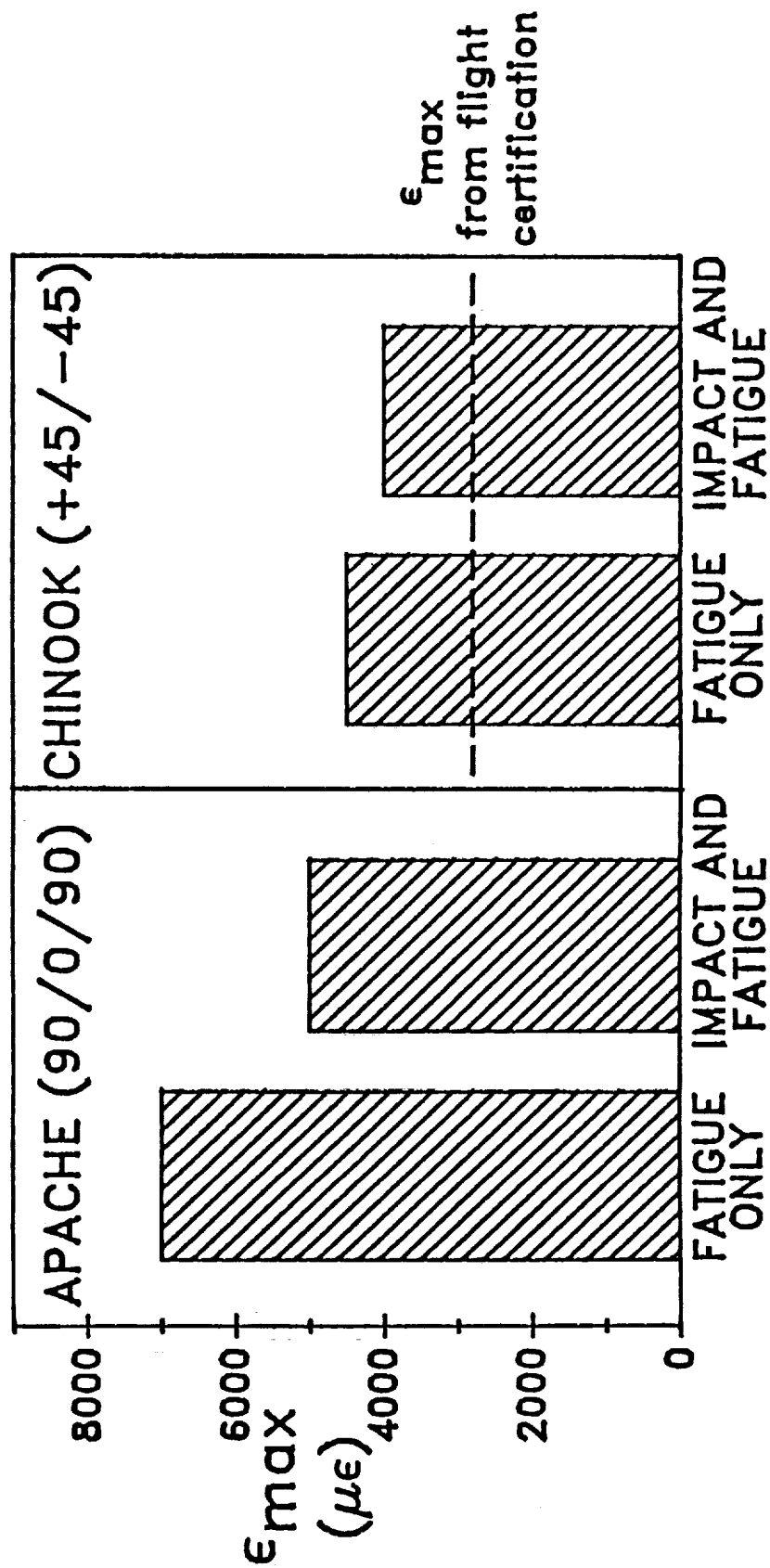


Figure 22(b).

EVALUATION OF THE RESILIENCY CHARACTERISTICS OF SEVERAL CANDIDATE
SOLID ROCKET BOOSTER ELASTOMERIC O-RING MATERIALS

C. L. Lach
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Ext. 43485 February 1989
RTOP 554-14-20
Code RM WBS 54-3

Research Objective: Characterize the resilient O-ring behavior in the Space Shuttle Solid Rocket Booster (SRB) field joint gland configuration for various temperatures and gap opening rates.

Approach: The Space Shuttle SRB's are assembled with elastomeric O-ring seals in the joints (see the left-hand figure). During the unique dynamic flexure conditions at launch, the O-rings are required to accommodate radial gap openings, axial gap openings, and pressure. Candidate O-ring materials were tested at temperatures from 20°F to 120°F, for various gap openings which simulated the 3-sigma pressurization to the joint and the external tank/SRB attach strut loads. The effects of gland orientation, grease condition, and initial compression were also addressed.

Accomplishment Description: The resiliency response, shown in the right-hand figure, decreased with a decrease in temperature and a corresponding increase in gap opening rate. The low frequency vibrations, a structural deflection resulting from the external tank/SRB attach strut loads had a negligible effect on the ability of the O-ring to track the gap opening. It was found that several of the elastomeric O-ring materials were detrimentally affected by the anti-corrosive grease used in the SRB field joints. However, the fluorocarbon elastomer, Viton V747-75, remained inert to the grease and was capable of accommodating the specified 0.018 in. gap opening (twice the maximum gap opening of 0.009 in.) at the required operating temperature of 75°F to 120°F.

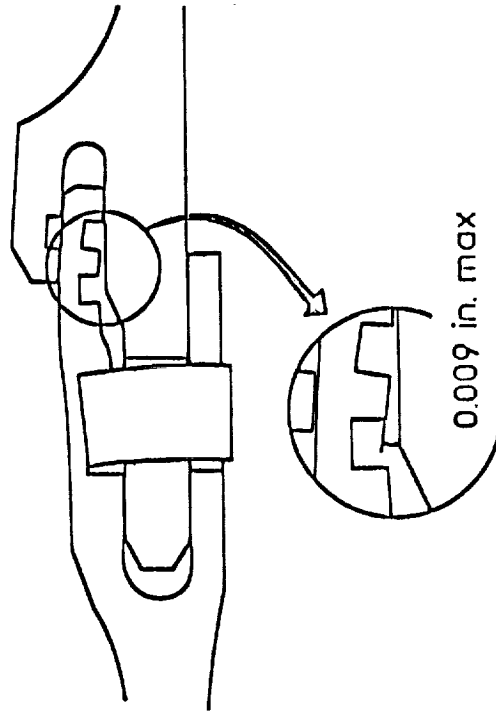
Significance: This study supported the redesign of the Space Shuttle SRB field joint by verifying the candidate materials against the design requirements and ultimately aided in selection of Viton, the final baseline O-ring material.

Future Plans: No further support, in elastomeric material testing, is proposed for the Marshall Space Flight Center SRB redesign program.

Figure 23(a).

SHUTTLE O-RING MATERIAL CHARACTERIZATION

Field Joint Gap Opening



Test 2(009)= 0.018 in

Viton (V747-75)

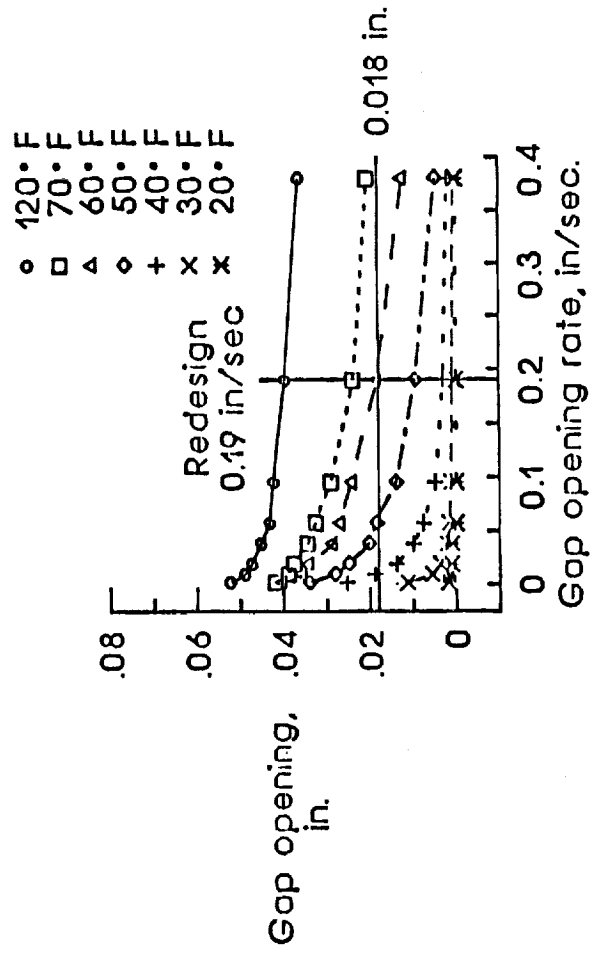


Figure 23(b).

EVALUATION OF O-RING GLAND SURFACE FINISH, CONTAMINANTS, AND GREASE BLOCKAGE ON THE SEALING PERFORMANCE OF THE O-RINGS DURING SIMULATED SPACE SHUTTLE LAUNCH CONDITIONS

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Mechanics of Materials Branch
Ext. 43485 August 1989
RTOP 505-63-01
Code RM WBS 54-3

Research Objective: Characterize the effects of surface finish, contaminants, and grease blockage on the sealing performance of the selected Space Shuttle O-ring material, Viton, in the redesigned Solid Rocket Booster (SRB) gland configuration.

Approach: The Space Shuttle SRB's are assembled with elastomeric O-ring seals in the joints. During the unique dynamic flexure conditions at launch the O-rings are required to accommodate radial gap openings, axial gap openings, and pressure [See Figure 24(b)]. Viton, the baseline O-ring, was tested at 75°F and at 120°F (the operating temperature limits), for the gap opening produced by a 3-sigma case of the head end motor pressurization of the field joint (1015 psi maximum). Low frequency vibrations caused by the external tank/SRB attach strut loads were superimposed onto the gap opening. Contamination effects on the sealing performance of the O-ring was evaluated using wire flaws, of 0.0005-0.004 inch diameters, placed across the primary O-ring to simulate human hair or fibrous debris. To verify the redesign leak check procedure, the grease wiping effect of the primary seal during SRB case assembly was simulated.

Accomplishment Description: The Viton O-ring sealed against all of the surface finishes (32 Ra to 250 Ra) from 120°F to 50°F. SEM observations confirmed abrasions found in O-rings that were leak checked and seal tested against the rougher finishes but were deemed noncritical as pressure leakages were not observed [See Figure 24(c)]. At 75°F and 120°F, verification of the leak check procedure utilizing the 1/4-scale bore seal fixture concluded that the threshold flaw size capable of detection through the grease blocked primary O-ring was 0.0016 inch diameter. Contaminants as small as 0.001 inch diameter were detected when the primary O-ring was not grease blocked.

Significance: This study supported the verification of the proposed leak check procedure for the redesign of the Space Shuttle SRB and confirmed the fact that flaws as small as 0.0016 inch in diameter can cause a failure to seal. A task force, aimed at incorporating a "clean room" environment for installation of the O-ring seals, was formed. The current relaxation in standards for the surface finish requirements does not result in the formation of detrimental leak paths.

Future Plans: No further materials testing is proposed for the verification of the redesign of the SRB in support of Marshall Space Flight Center.

Figure 24(a).

DYNAMIC FLAW SEAL TEST

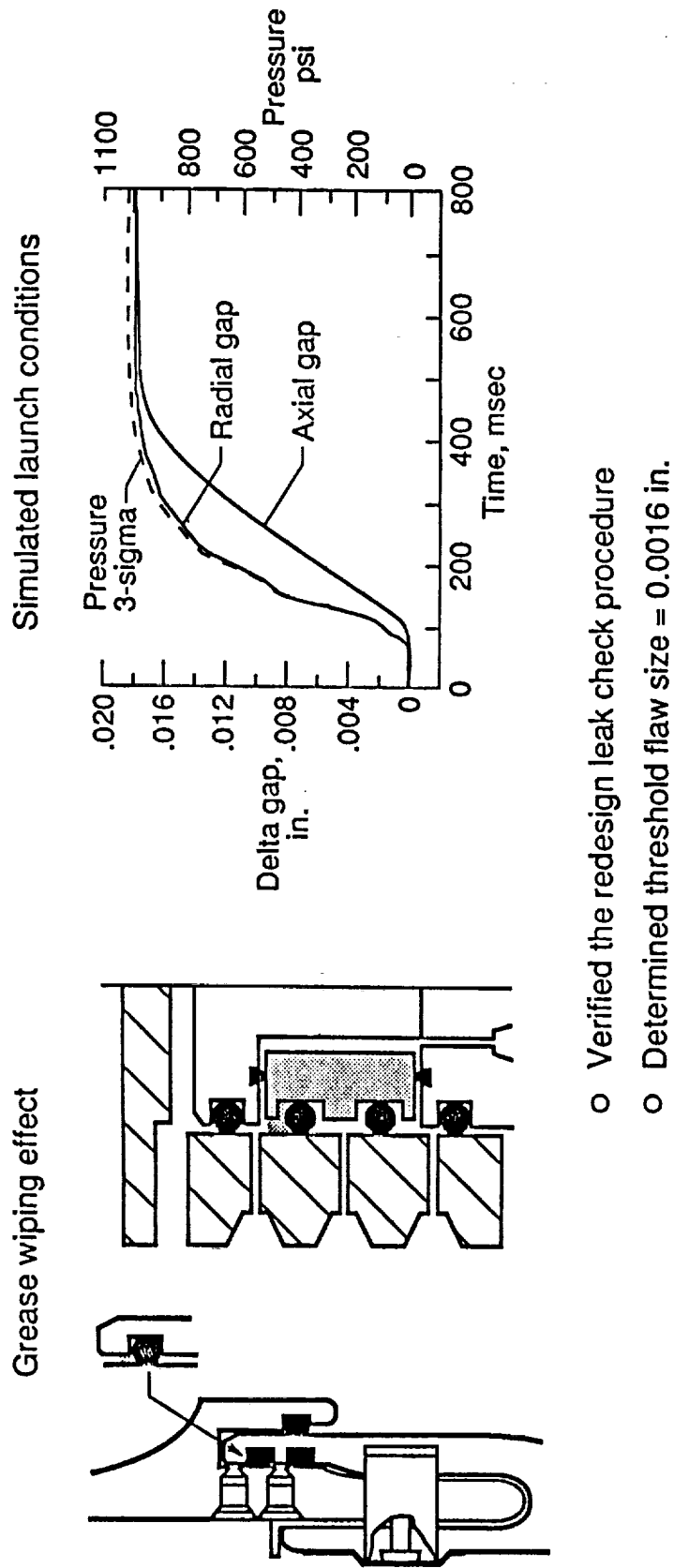


Figure 24(b).

SRB REDESIGN O-RING GLAND SURFACE FINISH STUDY

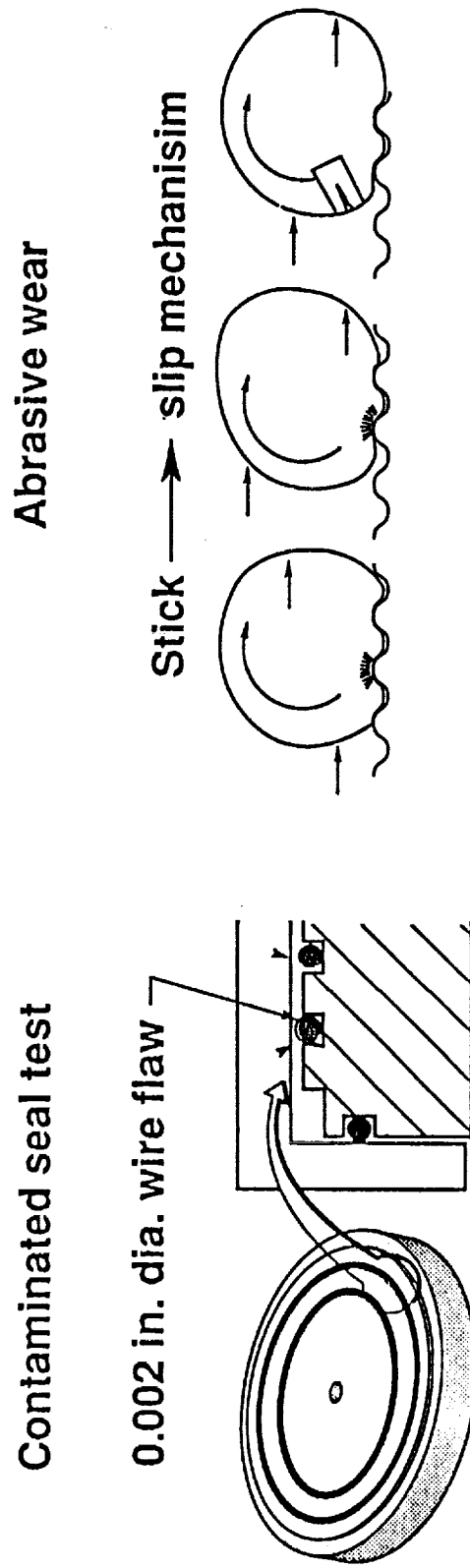
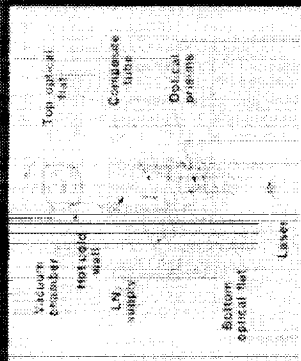


Figure 24(c).

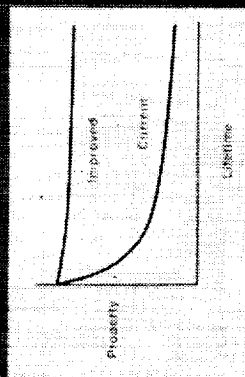
APPLIED MATERIALS

MATERIALS FOR SPACE STRUCTURES

DIMENSIONAL STABILITY

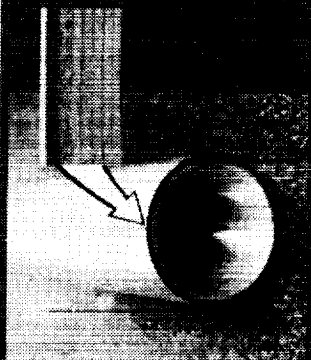


Laser Interferometry

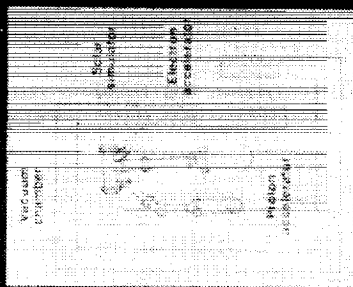


Durable Materials

PROTECTIVE COATINGS

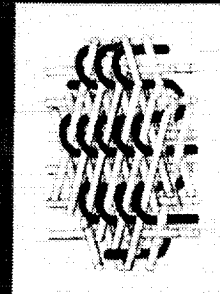


Coated Composite Tube

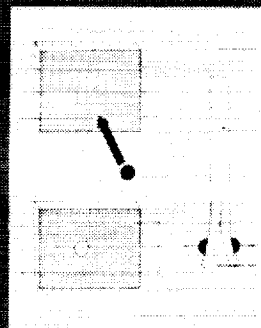


Radiation Effects

AIRCRAFT COMPOSITE MATERIALS

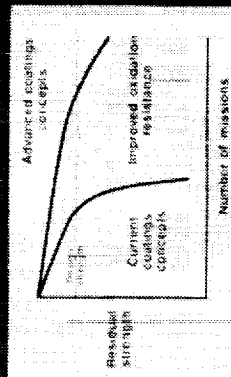
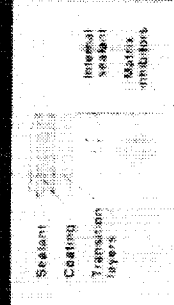


Material Forms and Processing



Material Testing and Analysis

CARBON-CARBON COMPOSITES



Oxidation Resistant Hot Aerostructure

Figure 25.

APPLIED MATERIALS BRANCH
FIVE YEAR PLAN

MAJOR THRUST	FY89	FY90	FY91	FY92	FY93	EXPECTED RESULTS
Space materials	Advanced space composites technology					Adv. composites concepts for future applications
	Precision segmented reflector					Precision reflector panels for LEO
	Global change initiative					Precision platform and reflector materials for LEO & GEO
	LDEF					Materials performance data base for extended exposures
Carbon-Carbon Composites	Carbon-carbon for NASP					C-C composites & coatings for NASP structure
	High strength, minimum gauge carbon-carbon					Strong, delamination resistant C-C for hypersonic vehicles
	Oxidation-resistant C-C concepts					Extended-life C-C for hot structure
Composite materials for aircraft and rotorcraft structures	Damage tolerant textile materials/processing concepts					Damage tolerant, cost effective materials for primary aircraft structure
	Innovative materials concepts					
	2D and 3D analytical modeling of advanced material concepts					Predictive capability for textile composites design

Figure 26.

TOTAL ABSORBED DOSE RATE EFFECTS FOR ELECTRON IRRADIATION OF ADVANCED THERMOPLASTICS

Edward R. Long, Jr. and Sheila Ann T. Long
Applied Materials Branch
Ext. 44249 May 1989
RTOP 506-43-21
Code RM WBS 54-2

Research Objective: Determine and explain the effects of total absorbed doses and absorbed dose rates on tensile properties for electron irradiation of advanced thermoplastic resins and composites.

Approach: Select a model thermoplastic material for study and expose it to electron radiation for varying total absorbed doses and dose rates. Determine the kinetics of molecular structural changes using electron paramagnetic resonance (EPR) data and relate the findings to the changes in the tensile properties of the material.

Accomplishment Description: An advanced thermoplastic material, a polyetherimide, has been exposed to 100-keV electron radiation for total absorbed doses of .001 MGy to 100 MGy at absorbed dose rates of .1 MGy/hr to 100 MGy/hr. Measurements were made of the material's tensile properties and radical densities before and after the exposures. The radicals' decay and creation kinetics were determined from the EPR data, and the results were correlated to the tensile elongation data to determine the damage threshold for the mechanical properties. As seen from the figure, the total dose threshold for large changes in the tensile elongation is approximately 2 MGy. There is no variation of the tensile properties with dose rates because the rate of radical creation exceeded the rate of radical decay (some of which results in crosslinking) for all the dose rates studied.

Significance: The data indicate that effects of laboratory exposures to electron radiation on the mechanical properties of the material are the same for dose rates from .1 MGy/hr to 100 MGy/hr and that a total absorbed dose threshold occurs at approximately 2 MGy. Below 2 MGy no measurable changes in mechanical properties occur, while above this dose significant decreases in elongation occur. Therefore, laboratory exposure dose rate is not as critical an issue as originally thought.

Future Plans: Conduct accelerated electron radiation exposures of carbon-fiber-reinforced thermoplastic composites to determine the effects of accumulated total absorbed doses for long-duration space missions in geosynchronous orbit.

Figure 27(a).

ELONGATION -TO- FAILURE OF IRRADIATED POLYETHERIMIDE FOR FOUR DOSE RATES

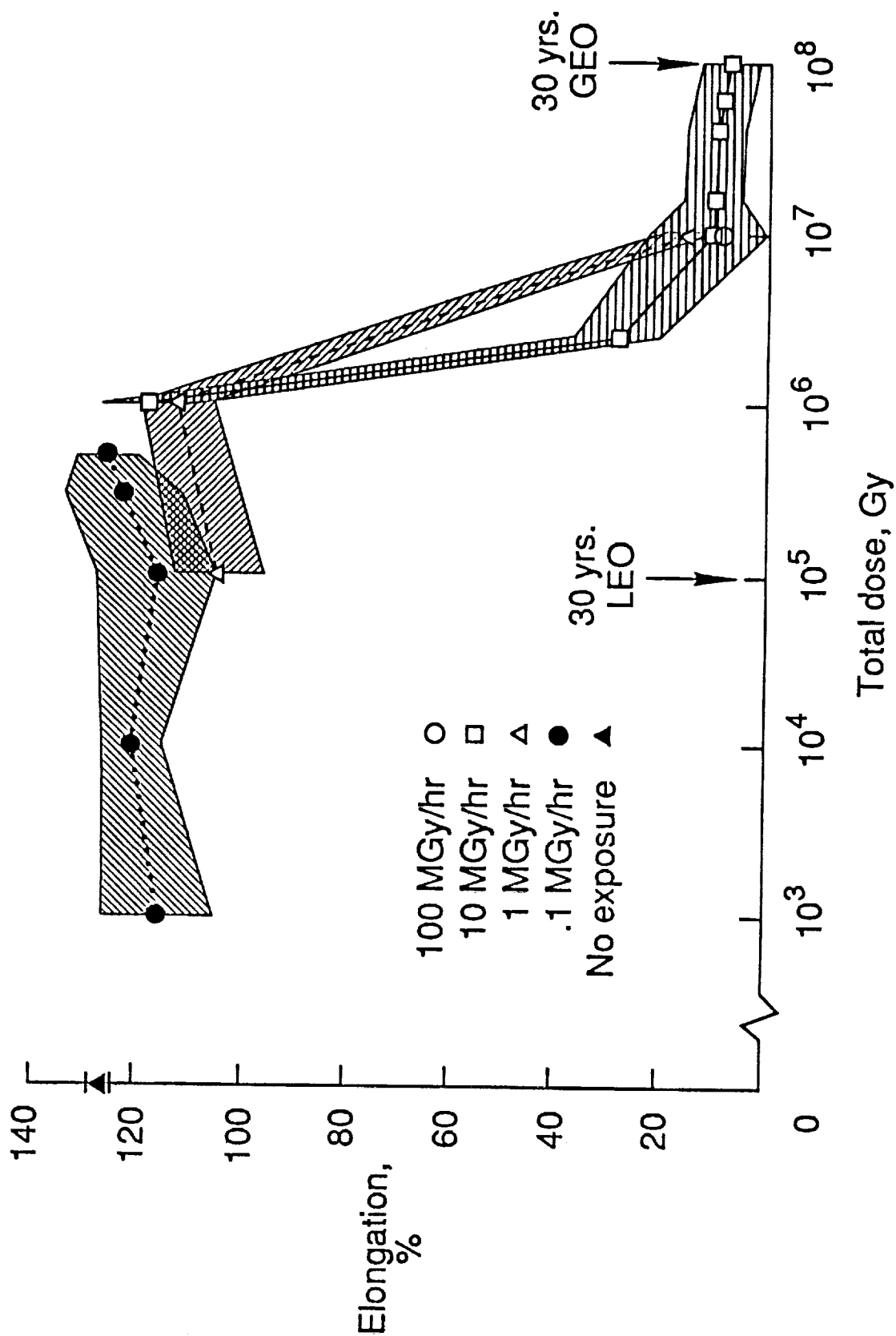


Figure 27(b).

AN INVESTIGATION OF FIBER SURFACE TREATMENT AS A MEANS OF IMPROVING INTERLAMINAR STRENGTHS OF CARBON-CARBON COMPOSITES

Howard G. Maahs and Y. Robert Yamaki (PRC)
Applied Materials Branch
Ext. 43498 January 1989
RTOP 506-43-71
Code RM WBS 54-2

Research Objective: Determine the extent to which the interlaminar strengths of carbon-carbon (C-C) composites can be controlled through surface treatment of the reinforcing fibers.

Approach: By chemically surface treating the carbon fibers employed as the reinforcement in C-C composites, it is possible to affect the bond strength between the fiber and the matrix material. Increasing this bond strength is one possible means of increasing the typically low interlaminar tensile (ILT) and interlaminar shear (ILS) strengths associated with C-C composites. But increases in bond strength could also further embrittle these composites, resulting in degradation of in-plane mechanical properties. In applications where ILT and/or ILS strengths are critical and some reduction in tensile strength is acceptable, the judicious use of fiber surface treatment may be a means of obtaining a more useful set of composite properties. To determine the extent of control over fiber-matrix interactions in C-C composites, phenolic-based unidirectional C-C composites were fabricated from a series of high-strength carbon fibers which had been surface treated by the manufacturer to levels ranging from 0 percent to 400 percent of the standard commercial level. The microstructures of these composites were examined and their ILT and ILS strengths were measured.

Accomplishment Description: Mechanical testing of the fabricated composites revealed a strong influence of fiber surface treatment on ILT and ILS strengths, as shown in figures 28(b) and 28(c), respectively. Peak or near peak interlaminar strengths were observed at a treatment level which was 50 percent of the commercial standard. This finding indicates that the treatment levels employed for commercially available fibers may be unnecessarily high for some applications in C-C composites, and that a significant degree of controllability exists for the treatment range 0 to 50 percent.

Future Plans: The present investigation focused solely on high-strength fibers because of the availability of these fibers. For the highest temperature applications of C-C composites, however, high-modulus (i.e., more thermally stable), rather than high-strength fibers, will likely be used. Follow-on plans to the present study involve determination of the mechanical properties of composites reinforced with high-modulus fibers similarly systematically surface treated to different levels. This study will also include measurements of in-plane tensile and compressive strengths and moduli. Longer range plans include the investigation of additives to the precursor matrix material as a means of influencing interfacial bond strength.

Figure 28(a).

VARIATION OF CARBON-CARBON INTERLAMINAR TENSILE STRENGTH WITH FIBER SURFACE TREATMENT LEVEL

Hercules Experimental Fiber

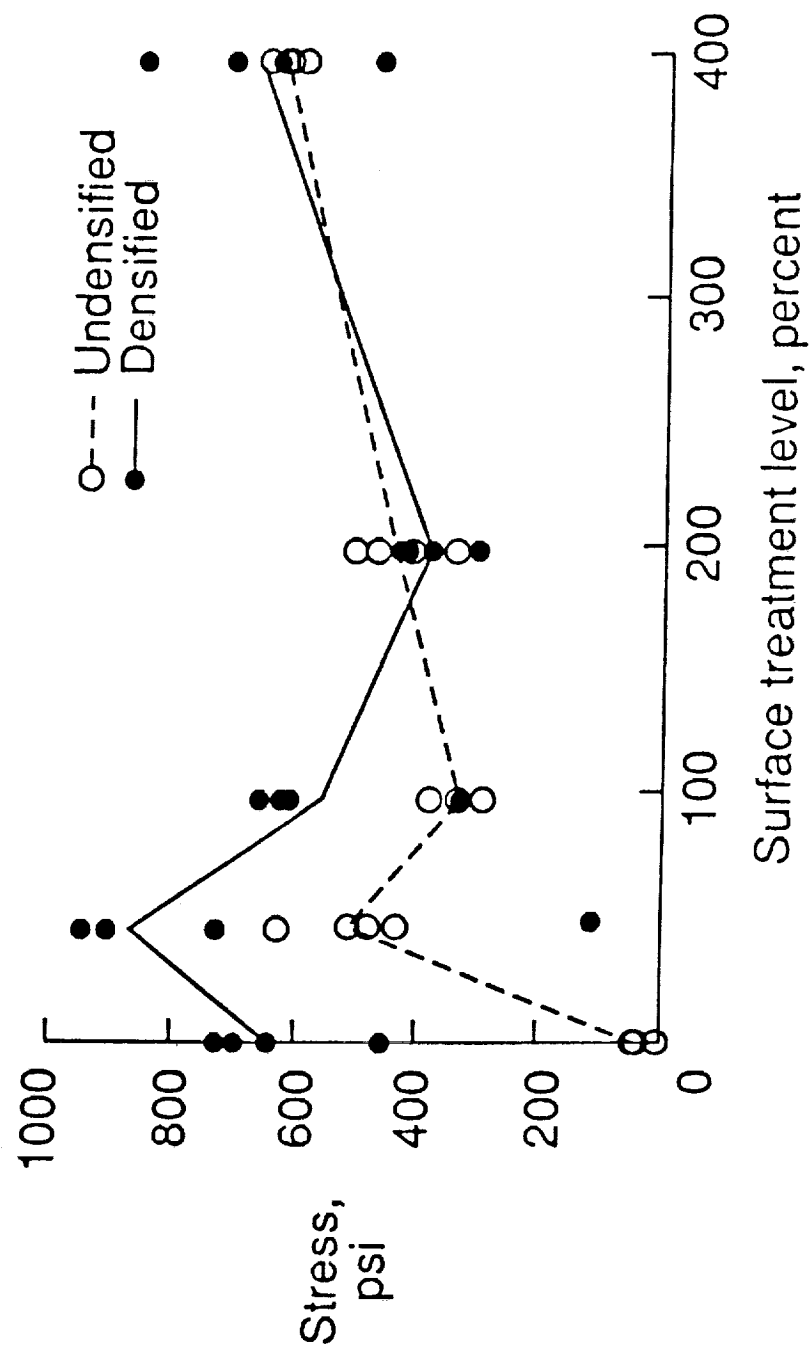


Figure 28(b).

VARIATION OF CARBON-CARBON INTERLAMINAR SHEAR STRENGTH WITH FIBER SURFACE TREATMENT LEVEL

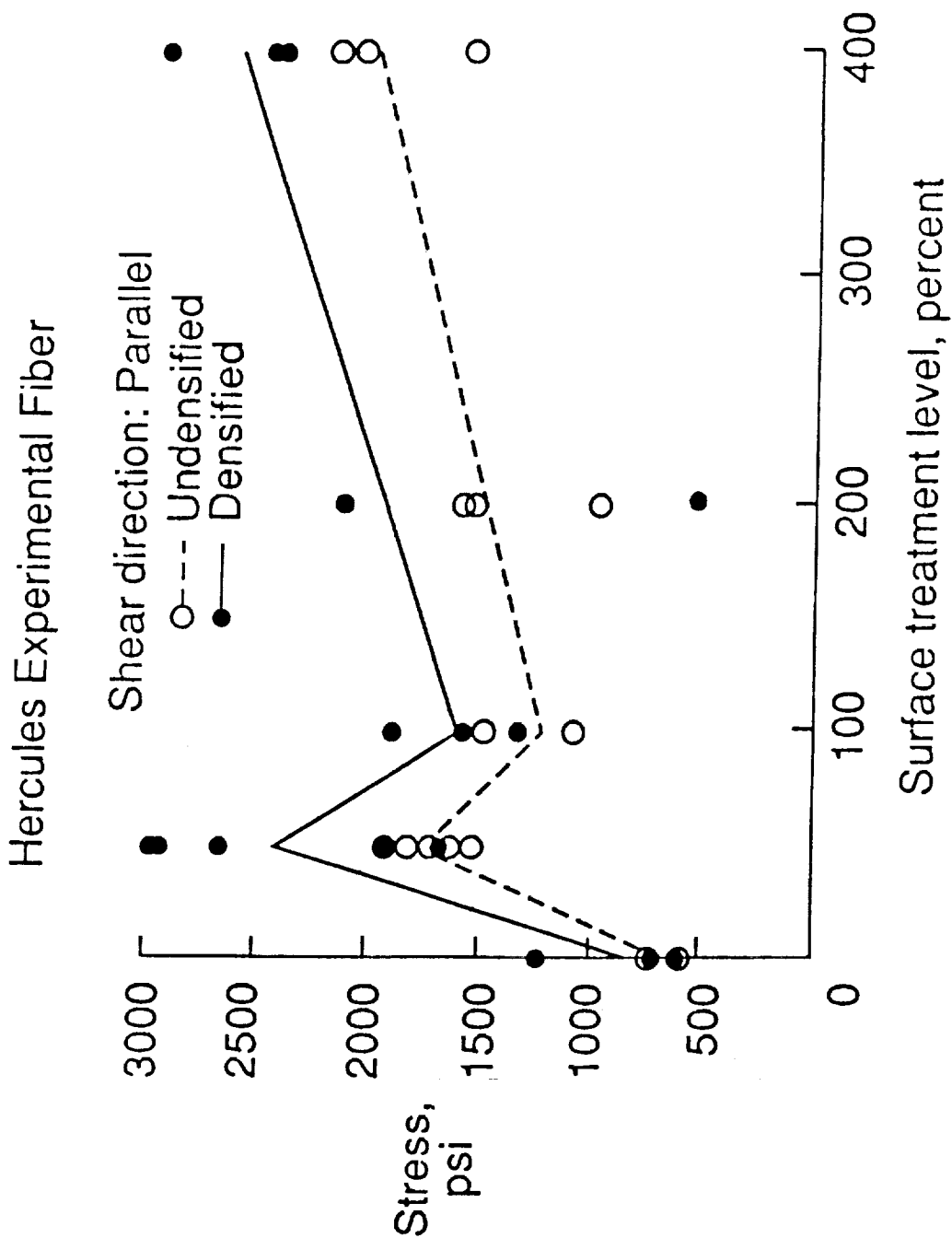


Figure 28(c).

EFFECT OF SURFACE MACHINING ON THIN 3-D ORTHOGONAL CARBON-CARBON COMPOSITES

Philip O. Ransone
Applied Materials Branch
Ext. 43503 March 1989
RTOP 506-43-71
Code RM WBS 54-3

Research Objective: Explore the potential and possible limitations of using 3-D orthogonal reinforcements to improve the interlaminar strengths of carbon-carbon (C-C) composites.

Approach: The use of through-the-thickness (z-direction) reinforcement has been demonstrated in previous work to be a very effective means of improving interlaminar properties of C-C composites. Interlaminar shear and interlaminar tensile strengths about twice those of typical 2-D composites have been achieved in composites reinforced with woven 3-D orthogonal preforms. However, several areas of concern exist. One is that the flexural moduli of 3-D C-C composites tend to be low relative to typical 2-D composites. Another concern is that the loop ends of the "z" reinforcing fibers present a rough external surface and removal of these loops may be desirable to achieve aerodynamic smoothness or to prepare the surfaces for accepting CVD coatings. Finally, the z-loops could, in any event, be destroyed during a conversion coating process for oxidation protection. Hence, a study was conducted to determine what effect removal of the z-loops has on mechanical properties.

Accomplishment Description: The z-loops were machined from the surface of 3-D C-C composites and mechanical properties were measured (see accompanying diagram). Interlaminar shear and interlaminar tensile strengths remained high, while flexural strengths and moduli were greatly improved, as illustrated in the plot of flexural strength vs. modulus. The reason for these higher properties is that the specimen thickness used in the stress calculations no longer includes the thickness contribution from the essentially non-load-bearing z-loops. Still, however, the flexural moduli are lower than the modulus for a typical 2-D composite such as ACC-4; this is because of the lower in-plane fiber volume fraction inherent in the 3-D fiber architecture.

Significance: Test results to date suggest that light machining of 3-D orthogonal C-C composites may be an acceptable approach for preparing surfaces for coating and to improve surface smoothness. Flexural strengths and moduli are, in fact, significantly improved. These results also imply that 3-D orthogonal C-C composites may possibly be conversion-coated without significantly degrading properties.

Future Plans: Conduct conversion coating trials on machined and unmachined 3-D composites. Refine the weave balance design between the number of z-yarns and in-plane yarns in 3-D orthogonal preforms to obtain the best combination of in-plane and interlaminar properties.

Figure 29(a).

EFFECT OF LIGHT SURFACE MACHINING ON FLEXURE PERFORMANCE OF THIN 3-D ORTHOGONAL CARBON - CARBON COMPOSITES

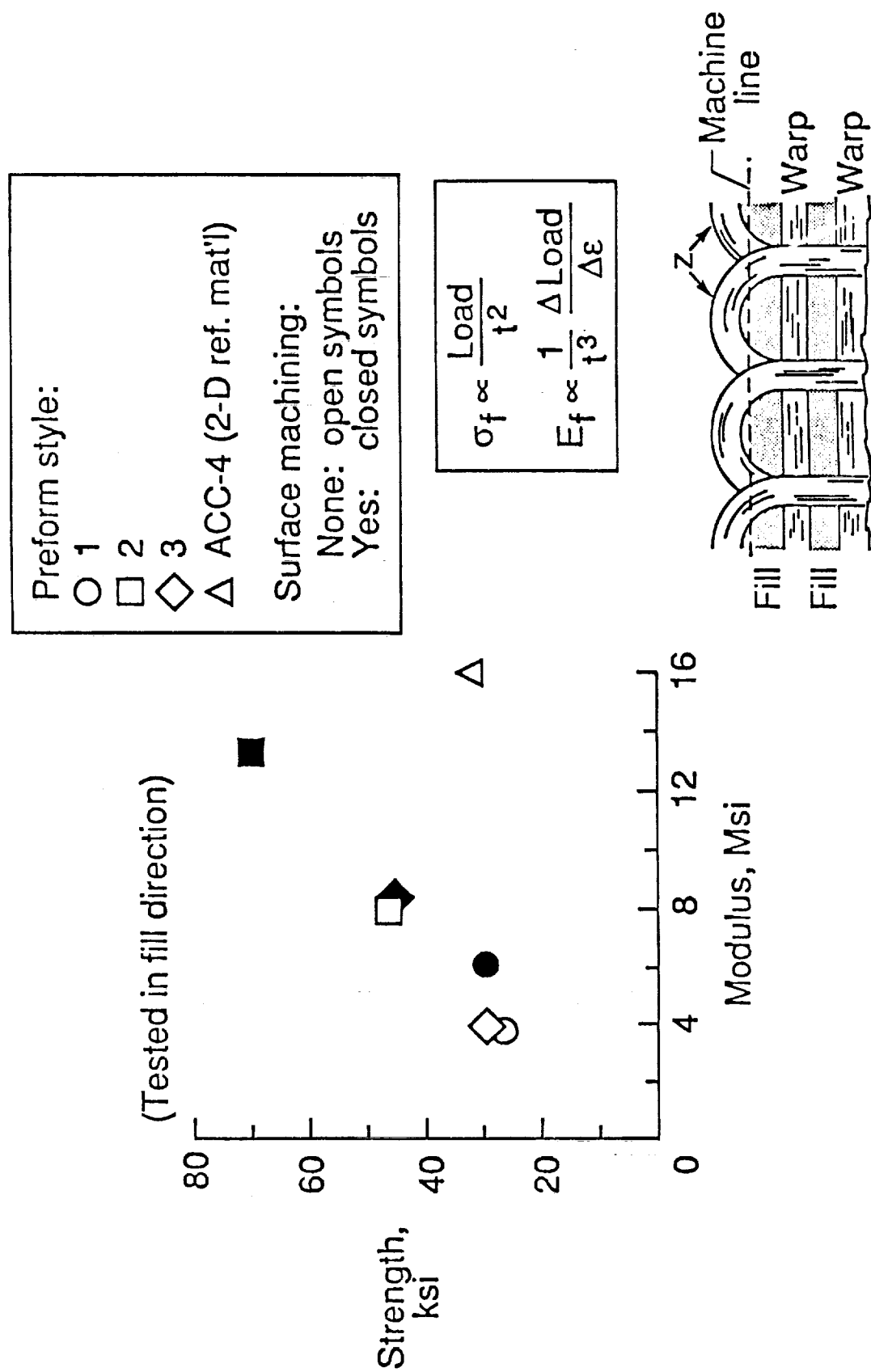


Figure 29(b).

EVALUATIONS OF OXIDATION-RESISTANT CARBON-CARBON COMPOSITES IN SIMULATED
HYPERSONIC VEHICLE ENVIRONMENTS

David M. Barrett
Applied Materials Branch
Ext. 43505 July 1989
RTOP 763-01-41
Code RM WBS 54-4

Research Objective: Identify major failure modes for oxidation-resistant carbon-carbon (ORCC) composites in hypersonic vehicle airframe environments, and identify major contributions to increased ORCC performance life.

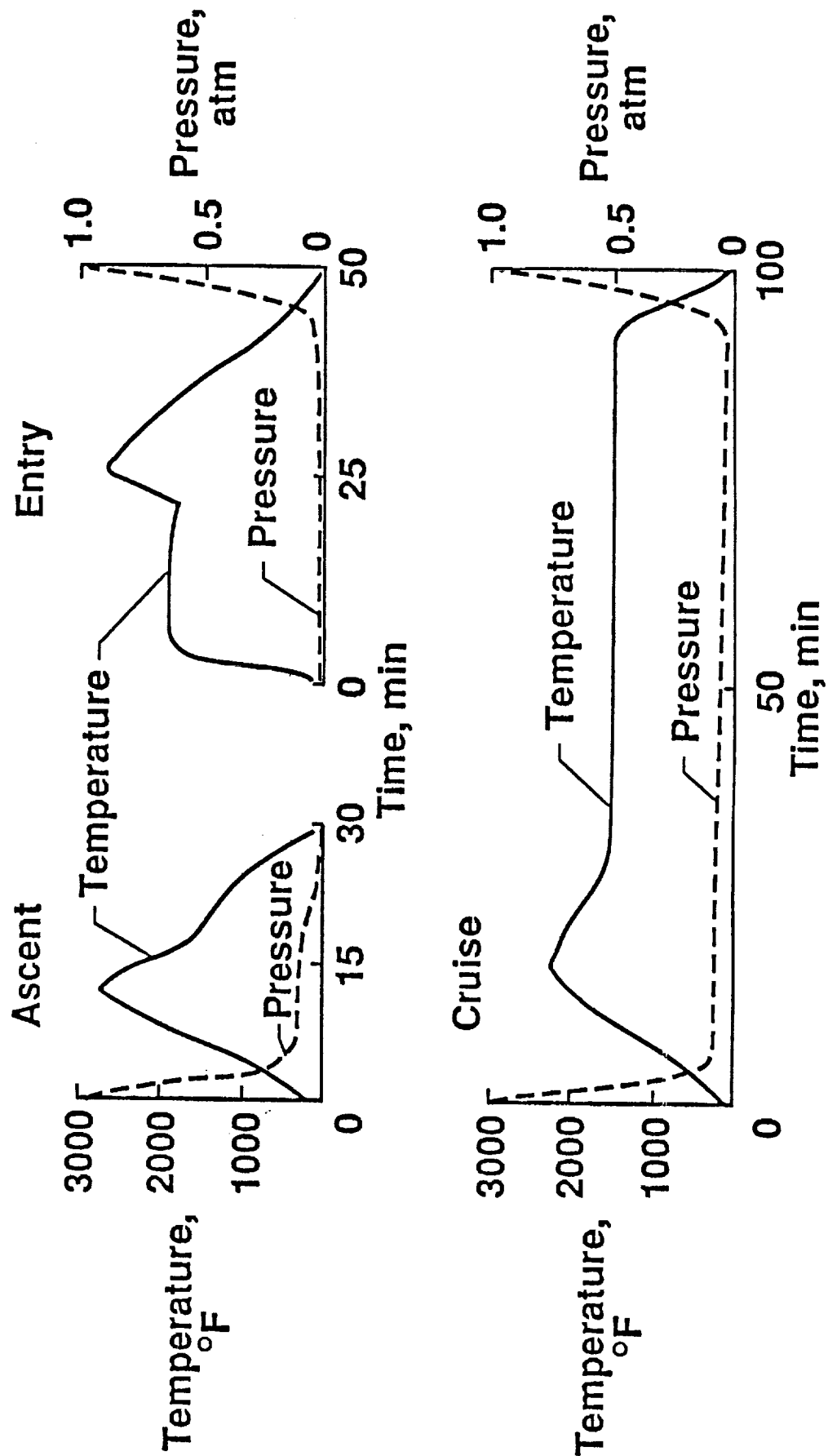
Approach: Select and test in simulated mission environments specimens of state-of-the-art ORCC composites protected with silicon-based coating systems. Conduct pretest and post-test analyses to determine the significant parameters affecting oxidation lifetimes.

Accomplishment: Specimens were oxidized in the Materials Division's multiparameter environmental simulators under conditions of temperature and pressure representative of those that might be experienced by an advanced aerospace vehicle designed to operate in both cruise and orbital modes. Figure 30(b) depicts time versus temperature and pressure profiles typical of those employed. A repeated sequence of these profiles, mixed with intermittent humidity exposures, was performed until the specimens lost 75 grams mass per square meter (roughly equivalent to a mass loss of about 2 percent for thin [0.1 inch] material). For these tests, a variety of specimens were obtained from LTV, SAIC, and CRT (Chromalloy Research and Technology). Since coating thickness varied among the specimens and even for different sides of the same specimen, coating thickness is an important parameter in determining performance as shown in the summary results in figure 30(c). The predominant failure mode observed for most specimens was coating separation. Another major finding is that boron in some concentration is essential for good coating performance. Also, there is no significant difference in performance between silicon nitride and silicon carbide-based coating produced by CVD; however, CVD coatings generally outperform straight conversion coatings. Additionally, overcoat glazes can have a very positive beneficial effect, and surface preparation is extremely important for good coating adherence.

Significance: These data indicate a fruitful direction to be taken to develop improved ORCC composites. Major parameters identified for particular attention are the boron-to-silicon ratio, the method of distribution of the boron within the coating, glaze composition, and surface preparation techniques.

Future Plans: Continue evaluations and performance analyses of other state-of-the-art ORCC materials. Investigate boron-doped conversion coatings and sol-gel-based coatings in-house. Initiate several research contracts to develop improved ORCC materials based on findings to date.

Figure 30(a).



Mission simulation test environment.

Figure 30(b).

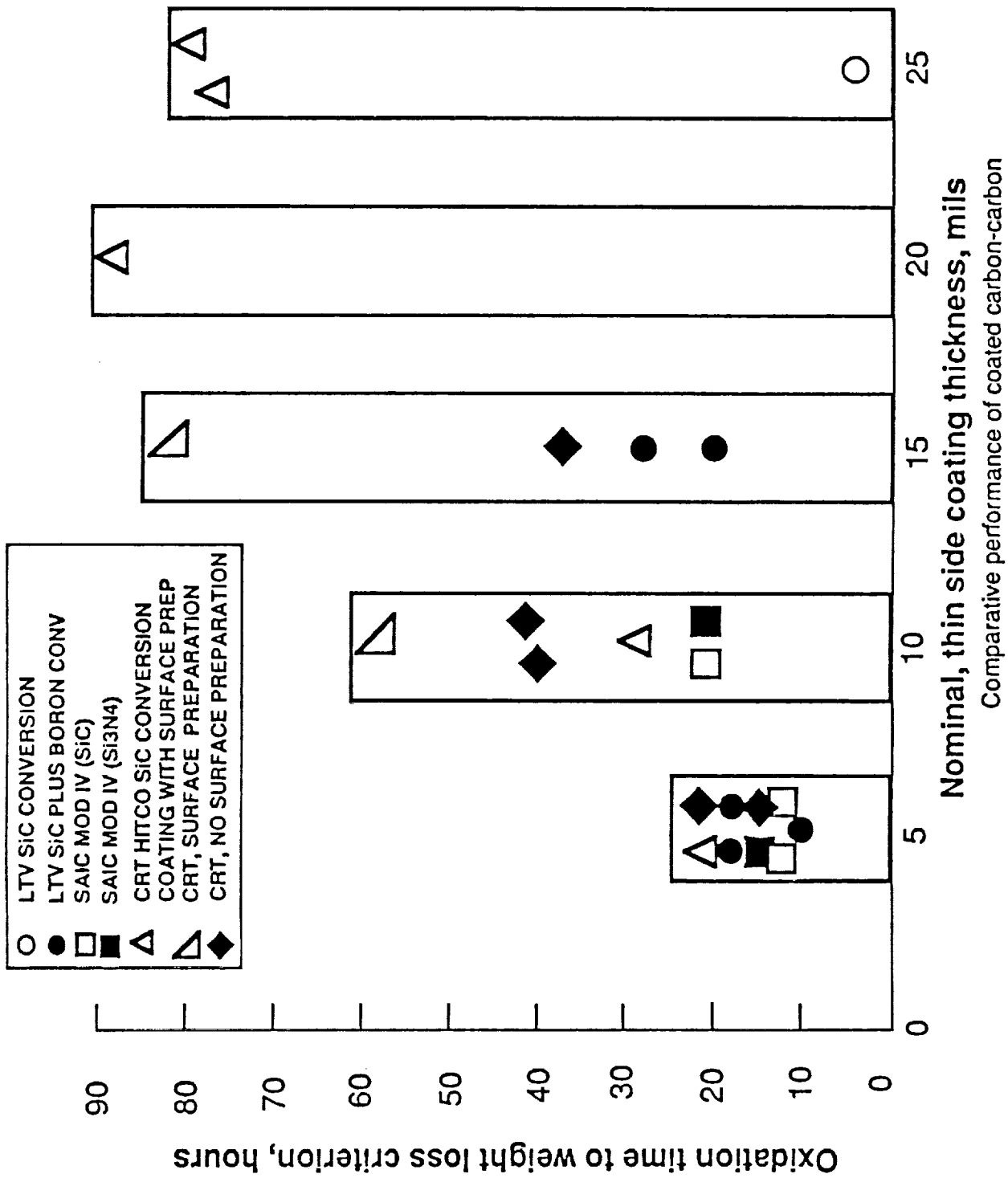


Figure 30(c).

RESIDUAL STRENGTH OF REPAIRED GRAPHITE/EPOXY LAMINATES
AFTER 5-YEARS OF OUTDOOR EXPOSURE

Jerry W. Deaton
Applied Materials Branch
Ext. 43087 November 1988
RTOP 505-63-01
Code RM WBS 55-1

Research Objective: To determine the environmental durability of repaired graphite/epoxy composite laminates.

Approach: Tensile tests were performed on a group of repaired graphite/epoxy specimens after 5 years of outdoor exposure to determine the effect of the outdoor exposure on the residual strength.

Accomplishment Description: The long-term durability of graphite/epoxy (Gr/Ep) repairs is being addressed in a 10-year outdoor exposure program at the Langley Research Center. The exposure specimen is a tabbed 16-ply Gr/Ep (T300/5208) laminate specimen that is 27 inches long and 8 inches wide as shown in figure 31(b). The damage consists of a 3.75 inch diameter hole. Four repair concepts are being evaluated and are applicable to secondary Gr/Ep structures for commercial transport applications. These include: (1) external bolted aluminum-plus adhesive, (2) precured bonded external Gr/Ep, (3) cure-in-place external Gr/Ep, and (4) cure-in-place flush Gr/Ep. Repaired specimens as well as undamaged and damaged unrepaired control specimens are being exposed outdoors (some unstressed, some under stress) for 10 years. Specimens are also being subjected to a 1/4 lifetime of tension/compression fatigue after each year of outdoor exposure. The residual tensile strength of unstressed, stressed, and full reversal fatigue specimens from each group have been obtained after 1, 3, and 5 years of outdoor exposure. The results for the unstressed [fig. 31(c)], stressed [fig. 31(d)], and full reversal fatigue [fig. 31(e)] specimens are compared with the tensile strength of baseline specimens which received no outdoor exposure. The baseline data indicate that the effectiveness of the repair concepts were: cure-in-place flush Gr/Ep, > 100 percent of baseline strength; cure-in-place external Gr/Ep, 79 percent; precured bonded external Gr/Ep, 67 percent; and bolted external aluminum, 55 percent. The data also indicate that the residual strengths for all repairs evaluated are essentially unaffected after 2 1/2 lifetimes of fatigue or 1 1/4 lifetimes of fatigue and 5 years of outdoor exposure.

Significance: These results indicate that strong, efficient repair concepts have been developed for secondary composite structures and that these repairs are essentially unaffected by 5-year outdoor environment plus 1 1/4 lifetime fatigue exposures.

Future Plans: Apply additional 1/4 lifetime of fatigue each year to fatigue group and perform residual strength tests after 7 and 10 years of outdoor exposure.

Figure 31(a).

RESIDUAL TENSILE STRENGTH OF REPAIRED GRAPHITE / EPOXY T300 / 5208 AFTER OUTDOOR EXPOSURE

Stressed at 22% ultimate

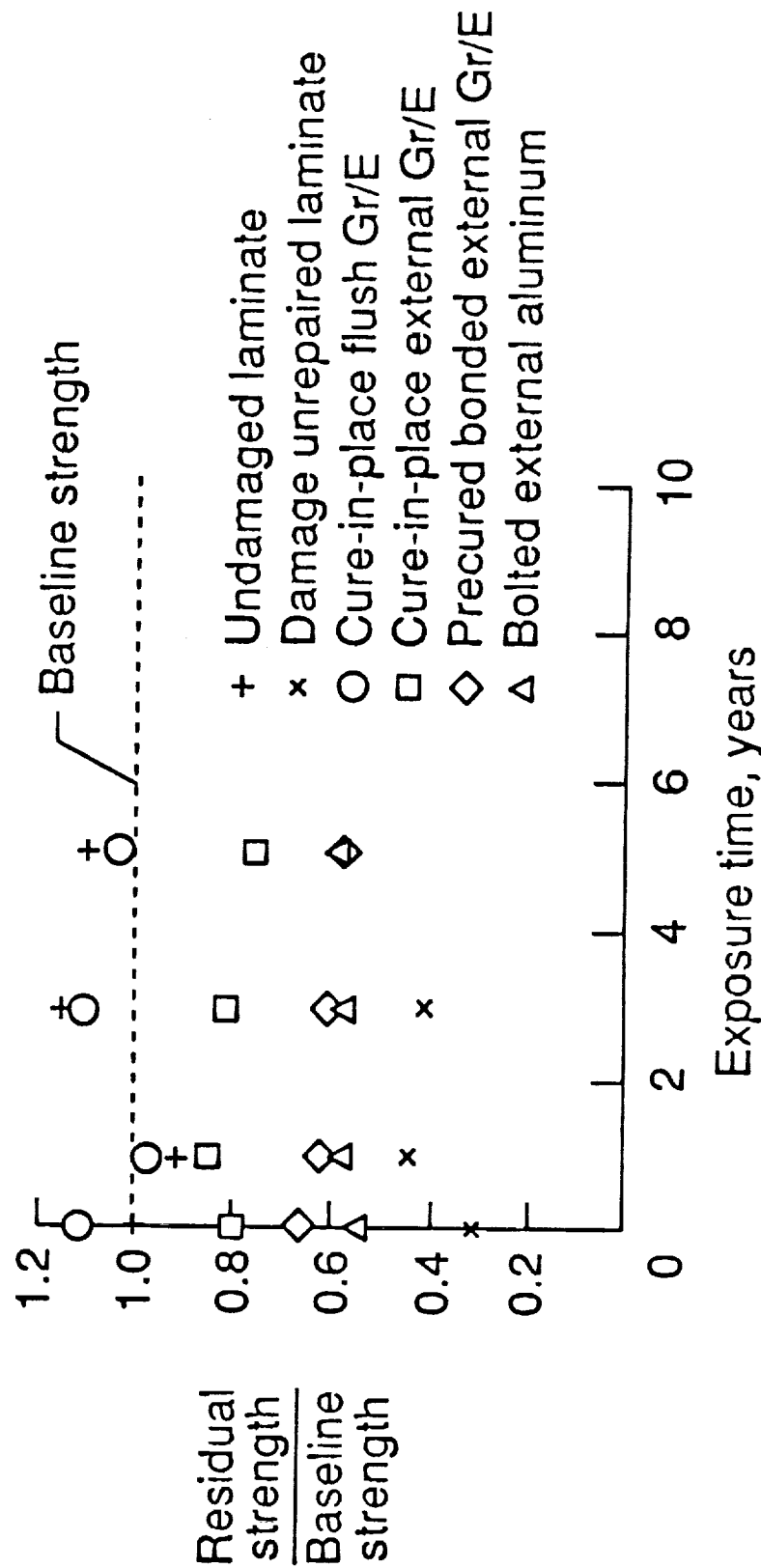


Figure 31(b).

RESIDUAL TENSILE STRENGTH OF REPAIRED GRAPHITE/EPOXY T300/5208 AFTER OUTDOOR EXPOSURE

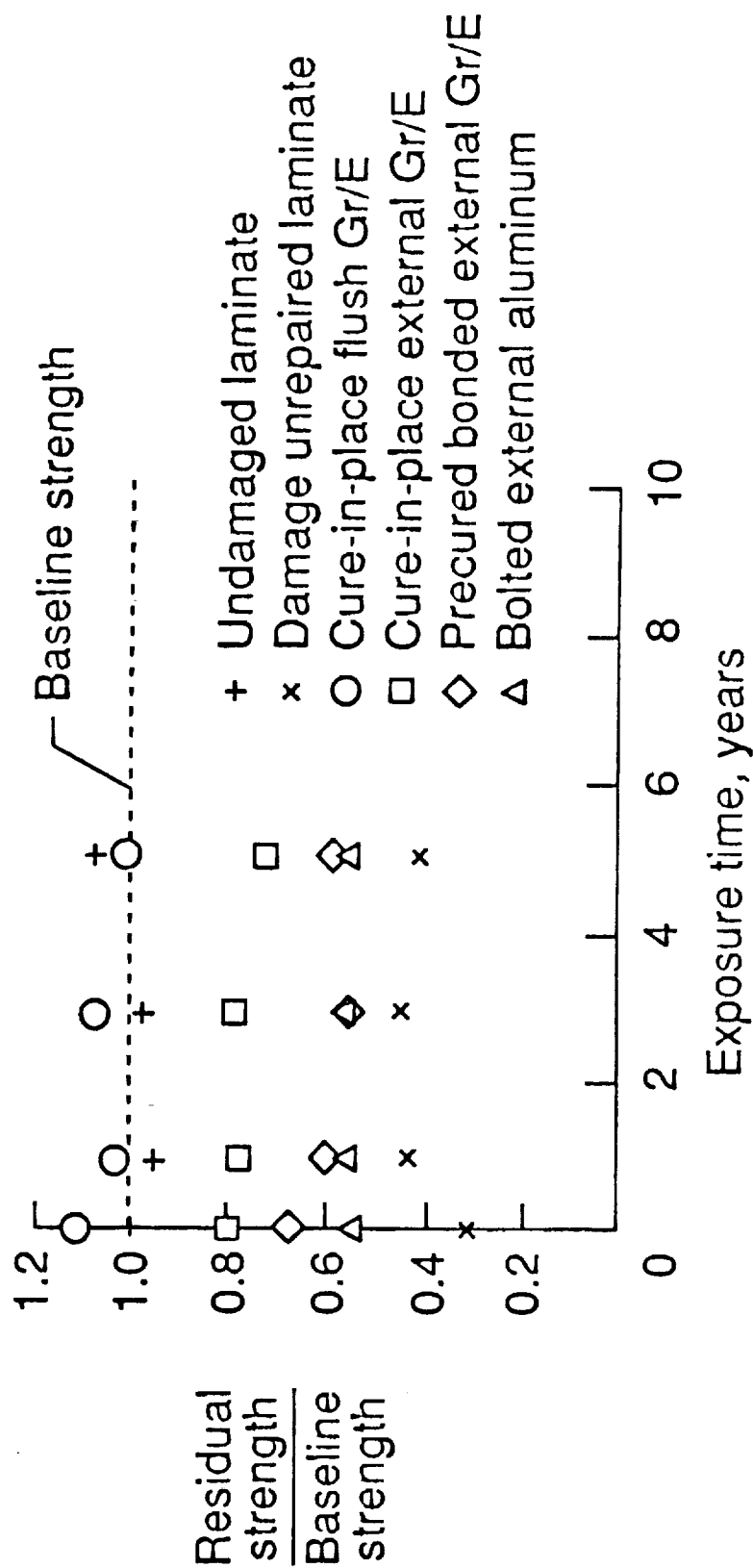


Figure 31(c).

TABBED LAMINATE EXPOSURE SPECIMEN

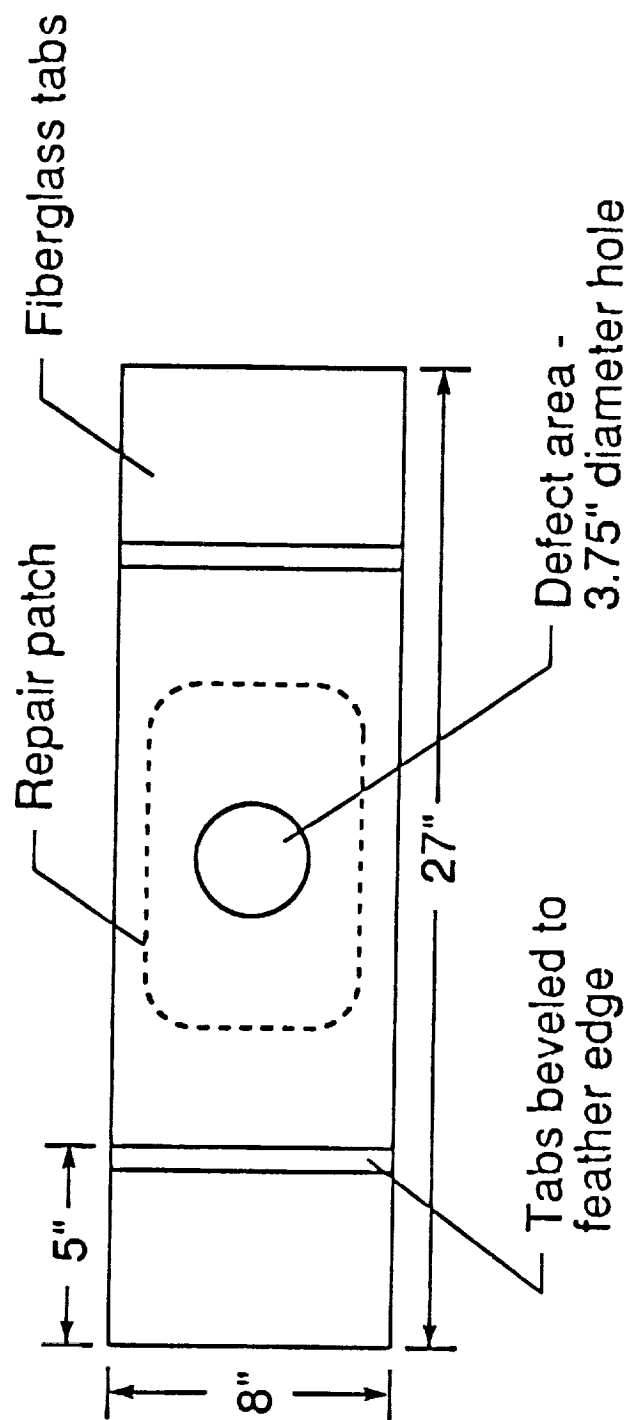


Figure 31(d).

RESIDUAL TENSILE STRENGTH OF REPAIRED GRAPHITE / EPOXY T300 / 5208 AFTER OUTDOOR EXPOSURE AND FATIGUE

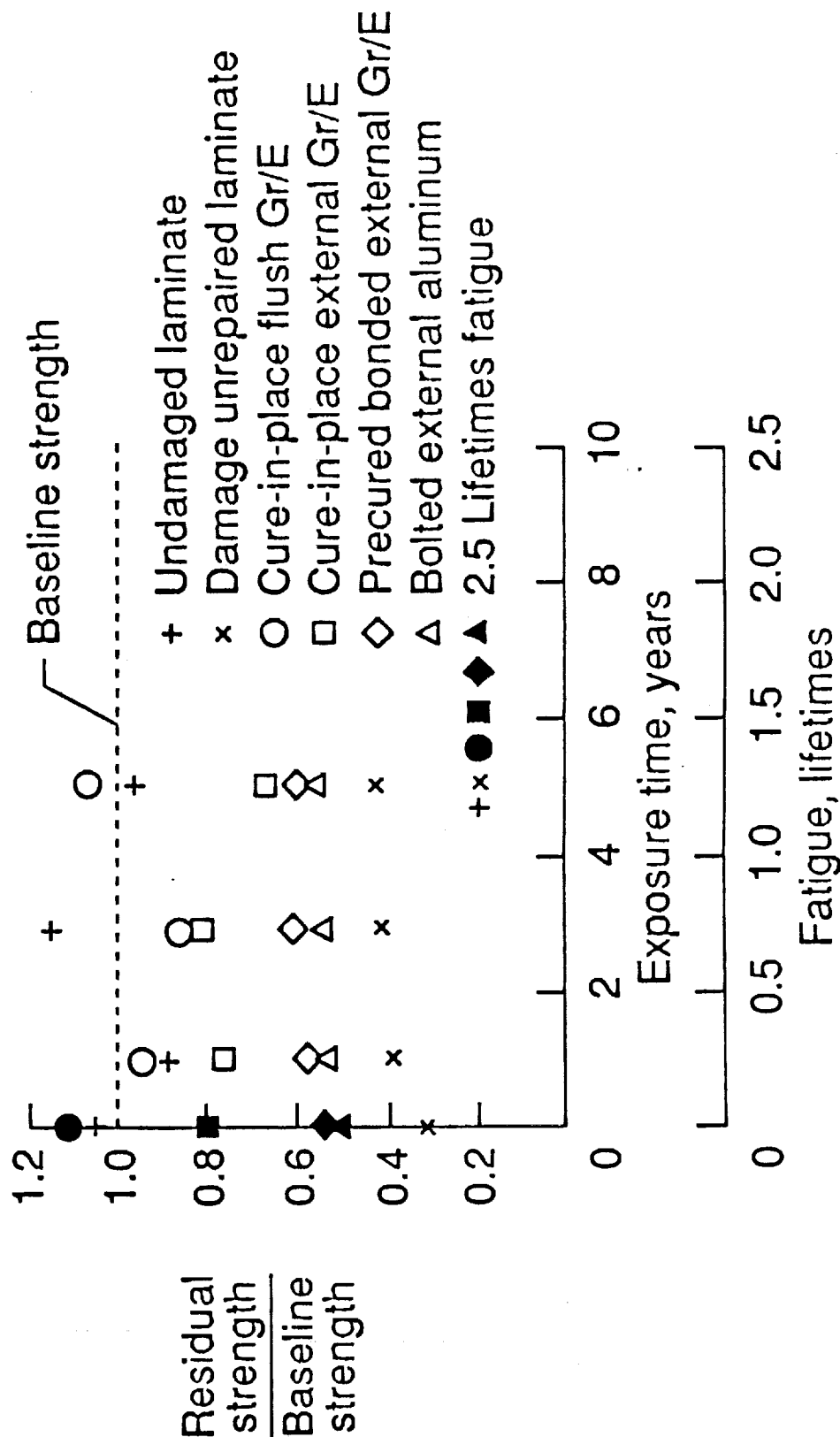


Figure 31(e).

IMPROVED CURE PROFILES FOR RESIN TRANSFER MOLDED CARBON-EPOXY COMPOSITE

Edward R. Long, Jr. and Alfred C. Loos*
Applied Materials Branch
Ext. 44249 September 1989
RTOP 505-63-01
Code RM WBS 52-1

Research Objective: Resin Transfer Molding (RTM) is a process used to consolidate advanced textile-preform composite materials for aerospace structures. The objective of this research is to develop an infiltration/cure processing model of resin transfer molded carbon-epoxy laminates and utilize the model to reduce the time required to achieve complete resin consolidation and cure.

Approach: Develop a computer model of the RTM process. For this model, experimentally measure required input viscosity data and differential scanning calorimetry data for Shell RSL 1282 resin cured with Epon 9470 hardener. Use the model with these input data to determine optimum dwell temperatures for laminate consolidation and predict resin cure advancement accounting for reaction exotherms during cure.

Accomplishment Description: An operational computer code has been developed. Using this code, an improved cure profile for the Shell resin has been determined which requires only one-half the time of the manufacturer's recommended cure profile. Figure 32(b) shows the manufacturer's time-temperature cure profile and the improved profile. The manufacturer's profile consists of temperature holds at 175, 250, 300, and 350°F; the total time, including cool down, is 350 minutes. In contrast, the improved profile consists of only two temperature holds, consolidation at 210°F for 60 minutes and cure at 350°F for 20 minutes; the total time, including cool down, is 175 minutes.

Significance: The processing model has provided a cure profile which saves both time and energy for resin transfer molding of fiber-reinforced epoxy laminates. The model can provide to the fabricator information on cure completion for panels of various thicknesses, for various fiber layout patterns, and for resin systems for which differential scanning calorimetry viscosity data are available.

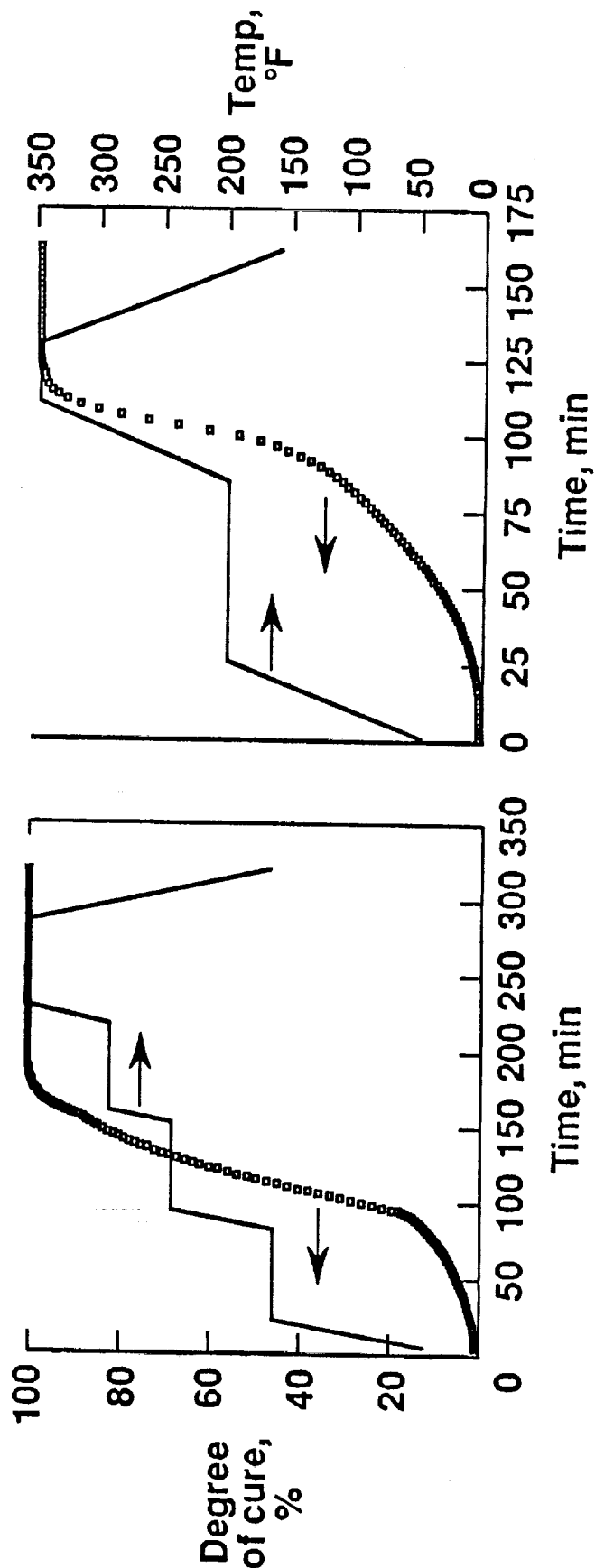
Future Plans: Verify the validity of the improved time-temperature cure profile for complete consolidation and cure. Compare laminate quality obtained from the improved and manufacturer's recommended profiles by mechanically testing and microscopically inspecting specimens cut from cured laminates.

*VPI&SU

Figure 32(a).

COMPARATIVE CURE PROFILES FOR RESIN TRANSFER MOLDED CARBON-EPOXY LAMINATES

— Time-temp cure profile
 Predicted degree of cure



Manufacturer's recommended
cure profile

Improved cure profile

Figure 32(b).

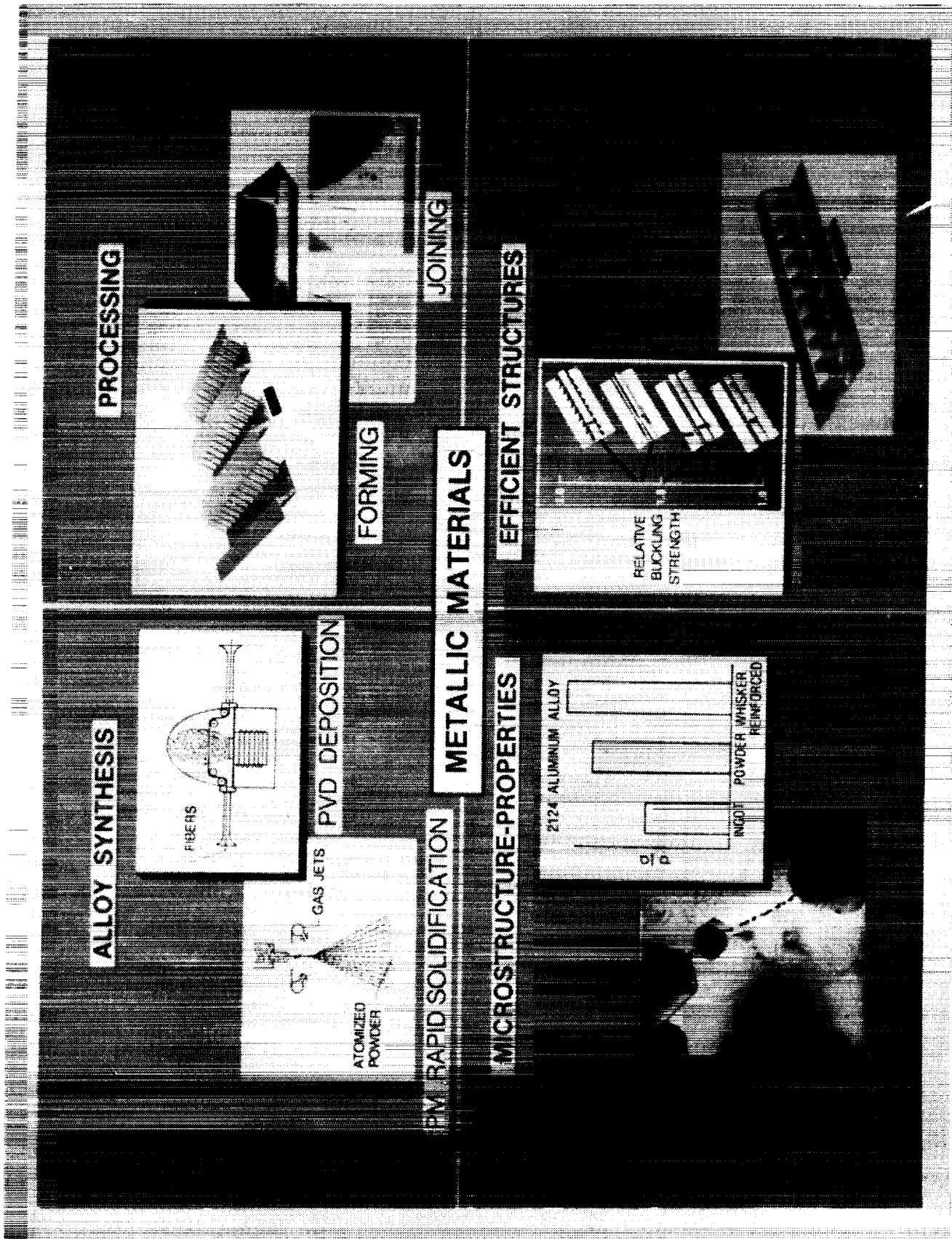


Figure 33.

METALLIC MATERIALS BRANCH
FIVE YEAR PLAN

MAJOR THRUST	FY90	FY91	FY92	FY93	FY94	EXPECTED RESULTS
Advanced light alloy and MMC development	PM aluminum alloys for high temp airframe and cryotanks					Improved metallics for transcentury and high speed transport aircraft and cryogenic tanks
	Aluminum lithium alloy technology					
	Development & characterization of aluminum matrix composites					
	Secondary and thermomech. processing effects on metallurgical structure & mechanical properties of light alloys and MMC					
Innovative metals processing	Aluminum alloy modifications for enhanced superplasticity & diffusion bonding studies					Processing and joining methods for lighter weight, lower cost aerospace structures
	Suppression and control of cavitation and determination of SPF parameters for Al alloys					
High temperature thin gage metals and MMC for airframe applications	SPF/Al and Ti alloy material/structural integration studies					Higher specific strength and stiffness materials for hypersonic vehicle airframes
	High temperature brazing/diffusion bonding studies of foil gage Ti and AMMC					
	Synthesis and characterization of thin gage high temperature metal matrix composites					
	Properties and stability of intermetallic alloy substrates by deposition					

Figure 34.

ENHANCED DIFFUSION BONDED Ti₃Al-Ti HONEYCOMB CORE SANDWICH PANELS

Eric K. Hoffman
Metallic Materials Branch
Ext. 43127 October 1988
RTOP 763-01-41
Code RM WBS 54-4

Research Objective: Develop joining processes for fabricating lightweight, high temperature sandwich structure using available titanium base model materials to provide a technology base leading to the fabrication of RSR Ti_xAl and Ti_xAl composite sandwich structure.

Approach: Conduct in-house studies using available titanium based ingot metallurgy (IM) model materials to develop joining processes suitable for fabricating Ti_xAl composite sandwich structures. Screen candidate joining processes and bonding parameters based on both metallurgical studies and mechanical property tests. Demonstrate fabrication technology readiness through design, fabrication, testing, and evaluation of sandwich specimens and structural sub-components. Verify suitability of the processing methodology for RSR and composite Ti_xAl materials as they become available.

Accomplishment Description: A process, termed Enhanced Diffusion Bonding (EDB), was developed to join the Ti-14Al-21Nb (Ti₃Al) face sheet, Ti-3Al-2.5V honeycomb core sandwich panel shown in the figure. Using a selective maskant and electroplating technique developed at LaRC, only the very edges of the honeycomb core were electroplated with EDB filler material prior to bonding. By limiting the placement of the EDB filler material to only the areas where it is needed, the structural weight and potential deleterious metallurgical interaction effects are minimized. The assembled panel components were then heated to 1700°F for a period of 30 minutes to promote bonding. Panels as light as 0.5 lb/ft² of panel area have been fabricated using this technique.

Structural element test results of EDB joints produced in Ti-14Al-21Nb alloy have demonstrated load carrying capability at temperatures higher than the 1700°F bonding temperature. As a result, the service temperature has the potential of being close to or equal to the bonding temperature. Consequently, EDB offers the potential of fabricating lightweight structural panels with high service temperatures relative to the bonding temperature.

Significance: NASP requires the development of advanced Ti_xAl intermetallic alloys having improved specific properties at elevated temperatures as well as joining processes capable of incorporating these materials into efficient structural components.

Future Plans: Alternate EDB material compositions and core materials will be evaluated in order to improve the elevated temperature mechanical properties of the Ti_xAl based sandwich panels. EDB sandwich specimens will be fabricated using cross plied Ti_xAl composite face sheets as they become available.

Figure 35(a).

ENHANCED DIFFUSION BONDED HONEYCOMB CORE SANDWICH SPECIMEN AND COMPONENTS

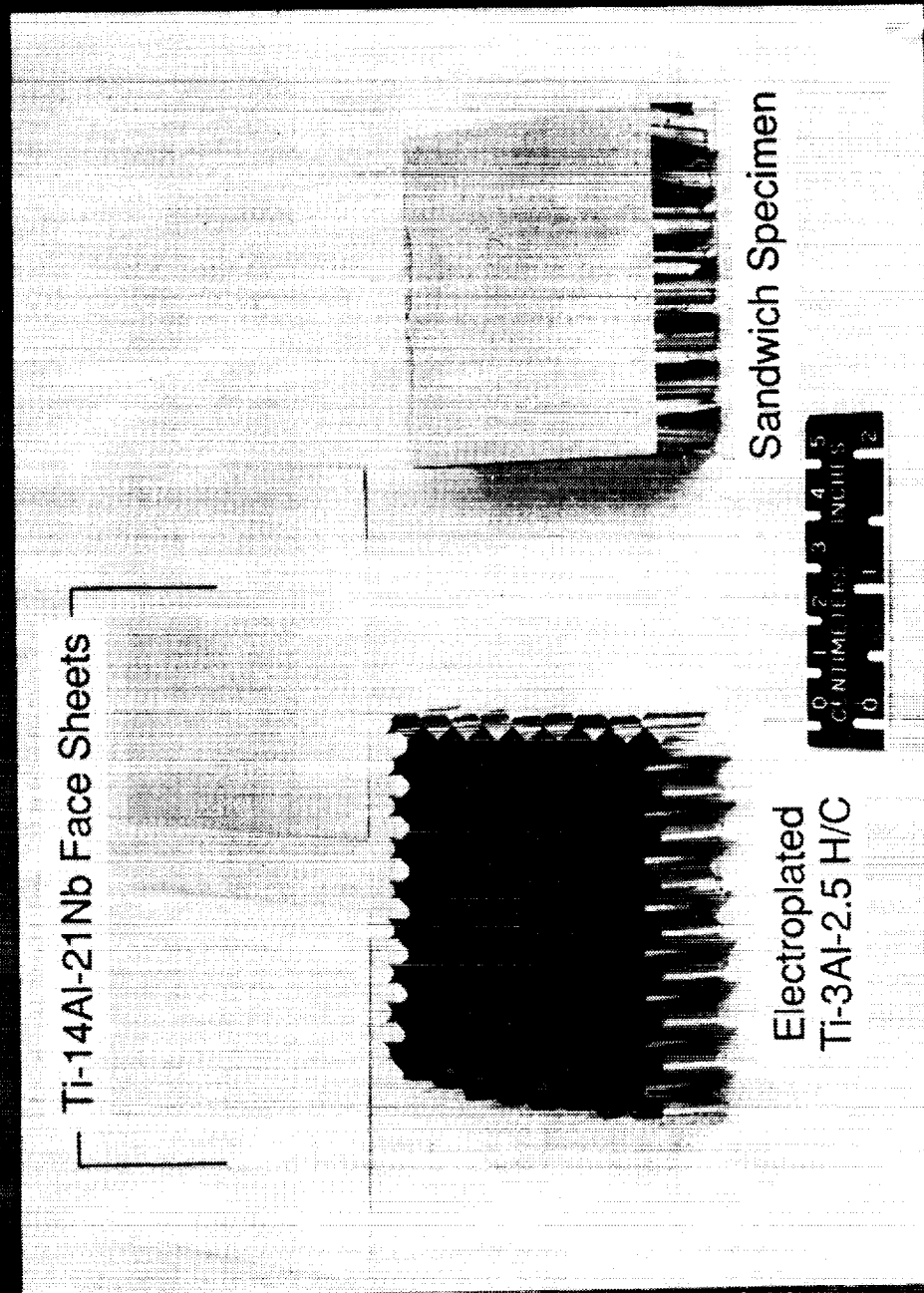


Figure 35(b)

DURABILITY OF GRAPHITE/EPOXY BOLTED JOINT SPECIMENS DEMONSTRATED
AFTER 10-YEAR EXPOSURE PROGRAM

Gregory R. Wichorek
Metallic Materials Branch
Ext. 43129 August 1989
RTOP 505-63-01
Code RM WBS 55-1

Research Objective: The establishment of a data base on the long-term durability of advanced composite materials for commercial aircraft applications through a ground-base environmental exposure program.

Approach: Augment the coupon exposure program at Langley with full-scale bolted joint specimens in order to provide long-term durability data on detailed joint designs subjected to environmental exposure over a 10-year period and to cyclic loading spectra typical of commercial transport flight service.

Accomplishment Description: Simplified splice joint specimens representative of side-of-body wing-skin splice in a commercial transport were designed and fabricated. The simplified splice joint specimens consisted of a single-row bolt configuration fabricated from T300/5208 and a double-row bolt configuration fabricated from T300/5209. The unpainted specimens were exposed to the outdoor environment, [Figure 36(b)], under a sustained tensile load and at yearly intervals subjected to 0.4 lifetime fatigue loading. At selected exposure times, tensile residual strength data were obtained for comparison with baseline data obtained from unexposed specimens.

Experimental results for the single- and double-row bolt configurations after 10 years outdoor exposure and 4 lifetimes of fatigue loading are shown in Figure 36(c). No significant reduction in residual strength occurred, although slight decreases in joint strength were observed after the first year environmental exposure. The T300/5208 specimens experienced slightly greater reductions in strength than the T300/5209 specimens. After 10 years of outdoor exposure under sustained tensile load and 4 lifetimes of fatigue loading, the T300/5208 specimen with the single-row configuration failed at design ultimate strength and the T300/5209 specimen with the double-row configuration failed above design ultimate load.

Significance: The results of this exposure program indicate that these graphite/epoxy materials when fabricated into full-scale components have good long-term durability for commercial aircraft applications.

Figure 36(a).

**OUTDOOR SUSTAINED-LOAD ENVIRONMENTAL TESTS ON
GRAPHITE/EPOXY BOLTED WING SPLICES**

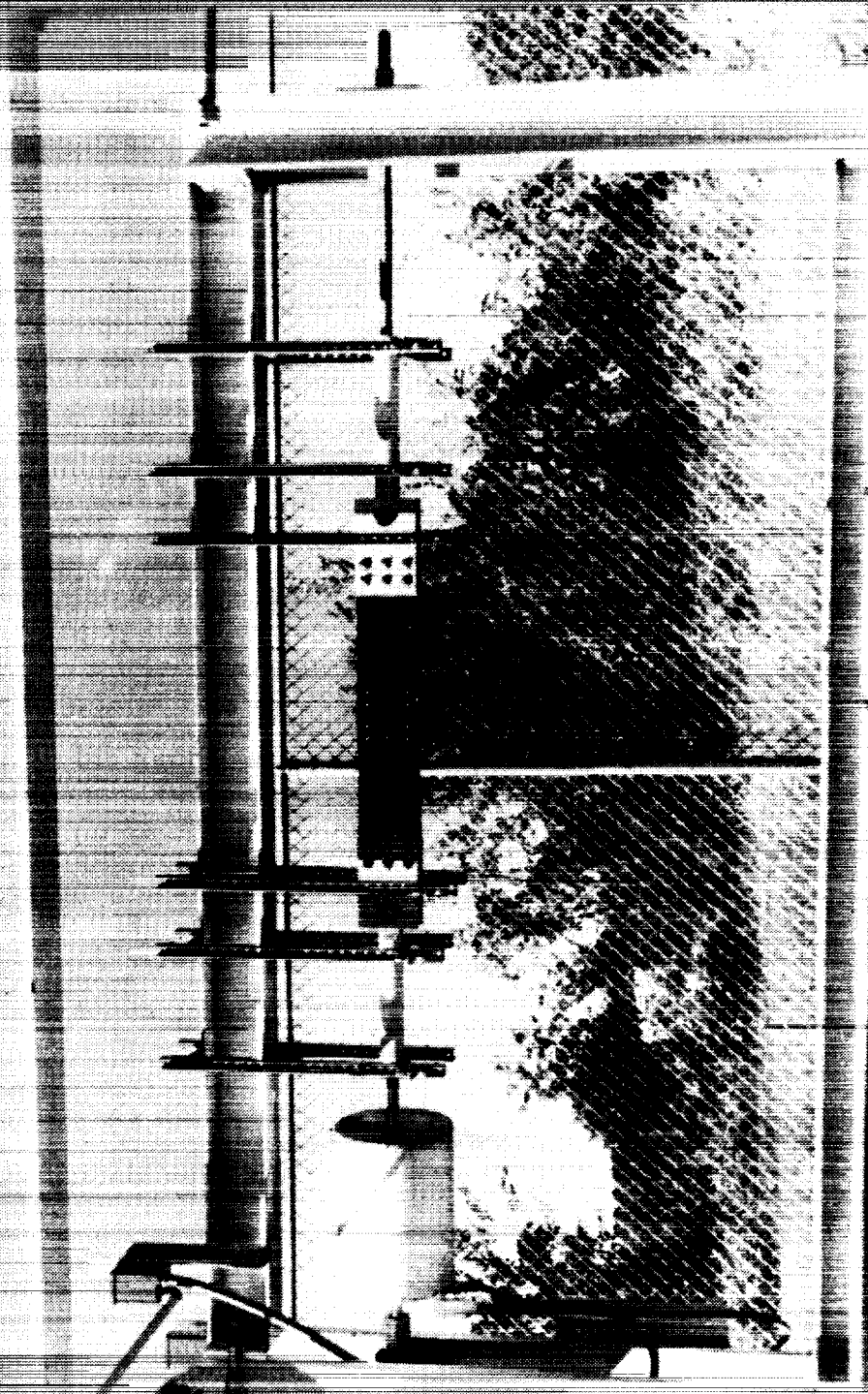


Figure 36(b)

EFFECT OF EXPOSURE AND LOAD HISTORY ON STATIC STRENGTH OF Gr/Ep BOLTED JOINTS

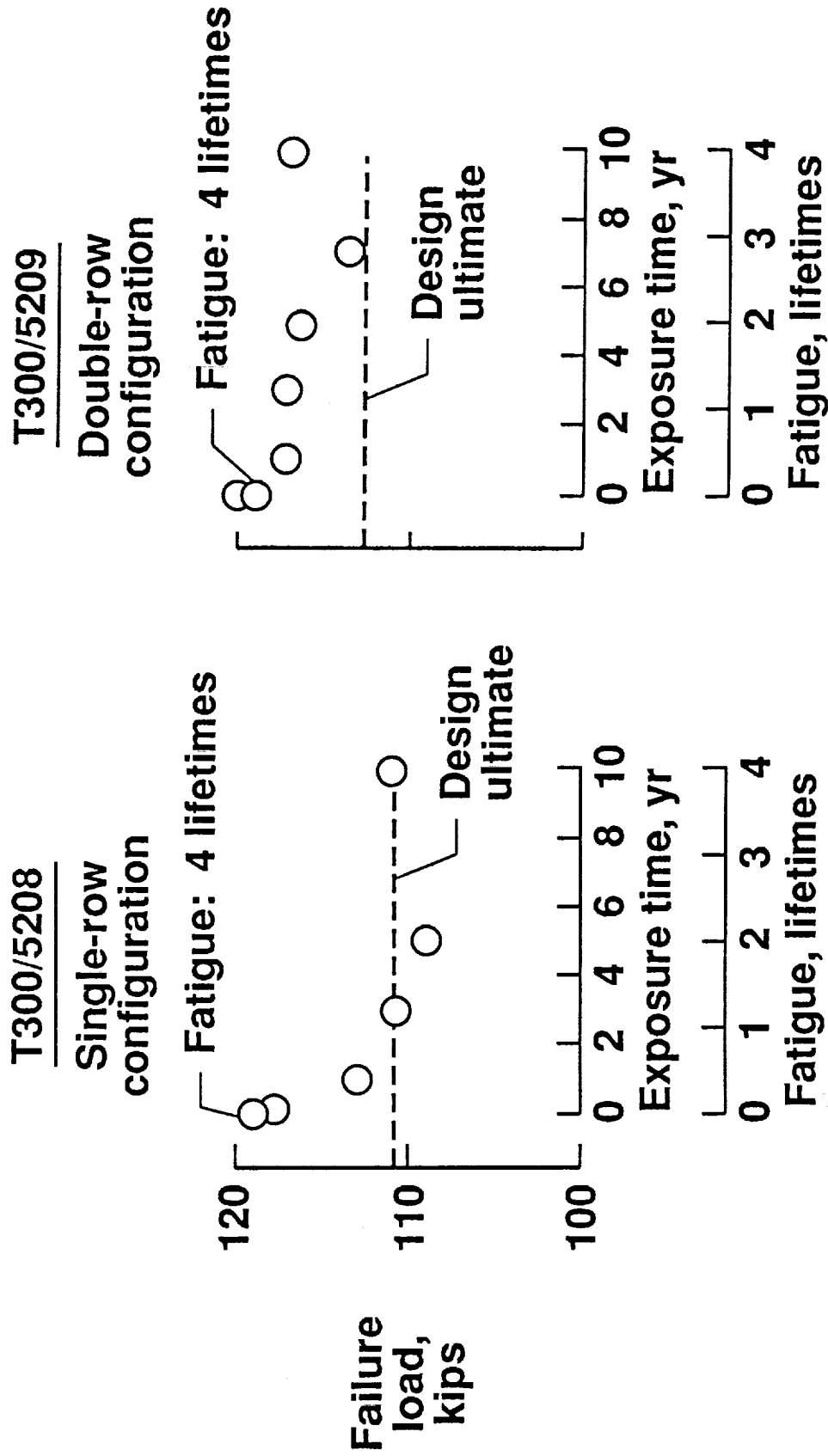


Figure 36(c).

SUPERPLASTIC FORMING OF ADVANCED ALUMINUM STRUCTURAL CONCEPTS PROMISE LIGHTER WEIGHT STRUCTURES

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Ext. 43125 December 1988
RTOP 505-63-01
Code RM WBS 52-3

Research Objective: Exploit the increased formability offered by superplastic forming to fabricate unique structural concepts for reducing the weight of future aerospace structures.

Approach: Conduct a comprehensive research program to determine the parameters for superplastic forming of promising aluminum structural alloys. Conduct analytical studies to identify candidate structural concepts. Fabricate and test small structural elements that depart from conventional shapes, and compare experimented results with analytical predictions.

Accomplishment Description: Superplastic forming parameters were established for fine-grained 7475 aluminum alloy. Tooling concepts and processing procedures for blow molding the aluminum sheet material into a mold cavity using inert gas pressure were developed. Based on the analytical studies conducted, a curved cap beaded web stiffener element was selected for fabrication and evaluation. Analyses predicted that the structural element selected offered a higher structural efficiency than a honeycomb-cone sandwich structure when loaded in compression. The figure shows the component following superplastic forming. The original thickness of the fine-grained 7475 aluminum sheet used to form the part was 0.020 inches and the part was formed at 960°F using a programmed inert gas pressure which correlated to a constant strain rate of 1×10^{-4} in./in./sec. Following forming, the specimen was trimmed and the ends were potted and machined for end compression testing. The test results obtained agreed favorably with analysis, demonstrating that the SPF does offer the potential to improve structural efficiency through the forming of uniquely shaped structural components which would be difficult or impossible to fabricate using conventional processes.

Significance: Results to date, on related contractual studies as well as in-house research, indicate that SPF can result in weight and cost savings of 30-50 percent for future aerospace structures.

Future Plans: Additional specimens of promising structural configurations will be superplastically formed and evaluated.

Figure 37(a).

**SUPERPLASTICALLY FORMED CURVED CAP
BEADED WEB STIFFENER**

7475 Aluminum Alloy

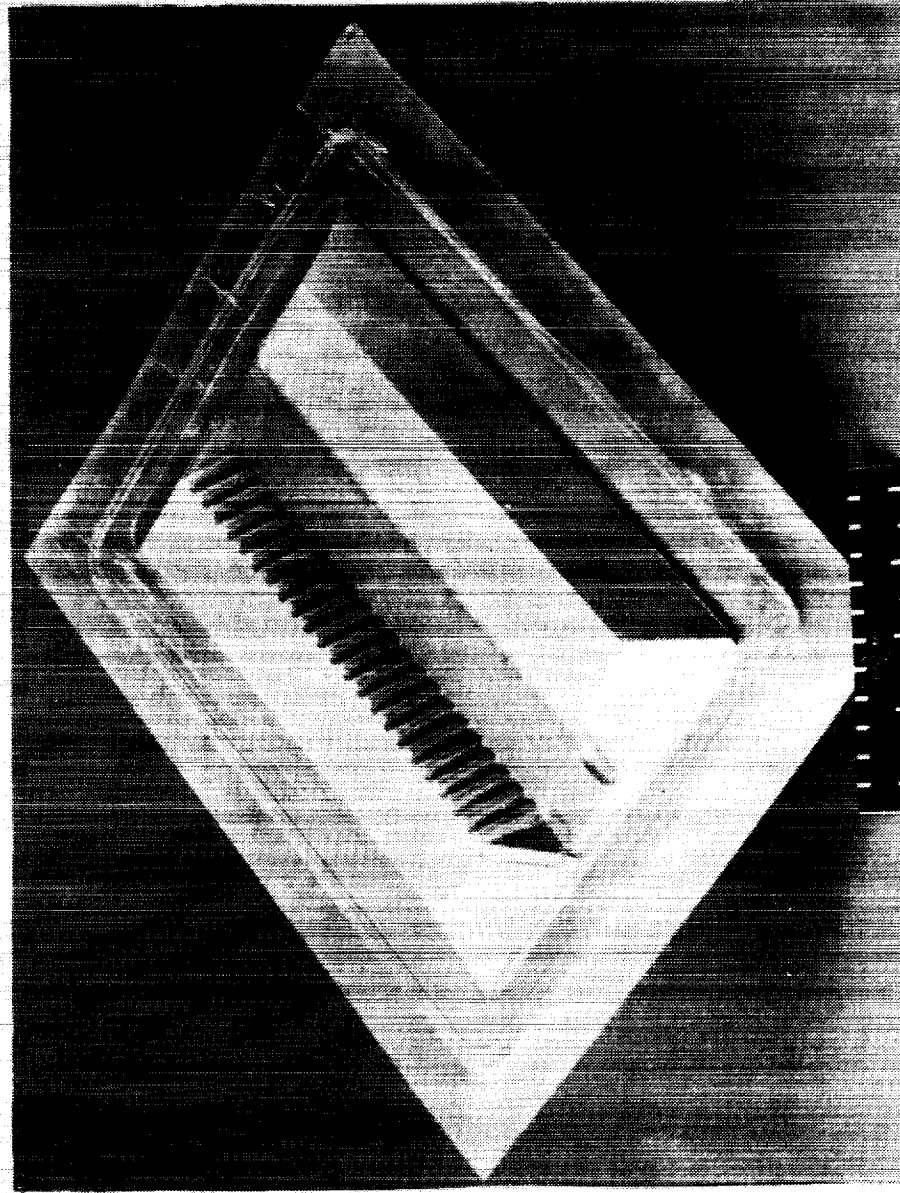


Figure 37(b)

COATINGS IMPROVE PERFORMANCE OF TITANIUM-ALUMINIDES

Terry A. Wallace, Karl E. Wiedemann and Dr. Ronald K. Clark
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Ext. 43511 February 1989
RTOP 506-43-71
Code RM WBS 54-4

Research Objective: Develop thermal control/oxidation preventive coatings for titanium aluminides in hypersonic vehicle applications.

Approach: Candidate coatings are identified and applied to various titanium aluminide substrates by a variety of coating techniques. Test samples are exposed to static oxidation environments and to simulated hypersonic conditions. The samples are analyzed to determine surface catalytic efficiency, emittance and oxidative characteristics.

Accomplishment Description: Coatings have been developed that produce high emittance, low catalysis surfaces on titanium aluminides that do not degrade during the simulated hypersonic exposure (Mach 3.7, 1800°F, 5 hrs.). Catalytic efficiency has been reduced by a factor of 25 while emittance has been increased by as much as 80% compared to uncoated alloys [Figure 38(b)]. Coatings that reduce the static oxidation rate of Ti₃Al by a factor of 10 have been developed [Figure 38(c)].

Significance: Titanium aluminide materials are prime candidates for use on hypersonic vehicles because of their high specific mechanical properties at elevated temperatures. However, the materials must be protected from the aero-thermal environment. High emittance, low catalysis coatings are required to reduce the net heat load to the structure by increasing the heat radiated from the surface and by reducing the heat input from the recombination of dissociated gas species present in the hypersonic environment. These new protective coatings will allow titanium aluminides to reach their full potential as structural materials for hypersonic applications.

Future Plans: The most promising coatings will be combined into multilayer coating systems to provide total environmental protection. Coatings will be modified as required for compatibility with new alloys.

Figure 38(a).

COATINGS IMPROVE PERFORMANCE OF TITANIUM-ALUMINIDE

Hypersonic Flight: 5 Hr 1800°F

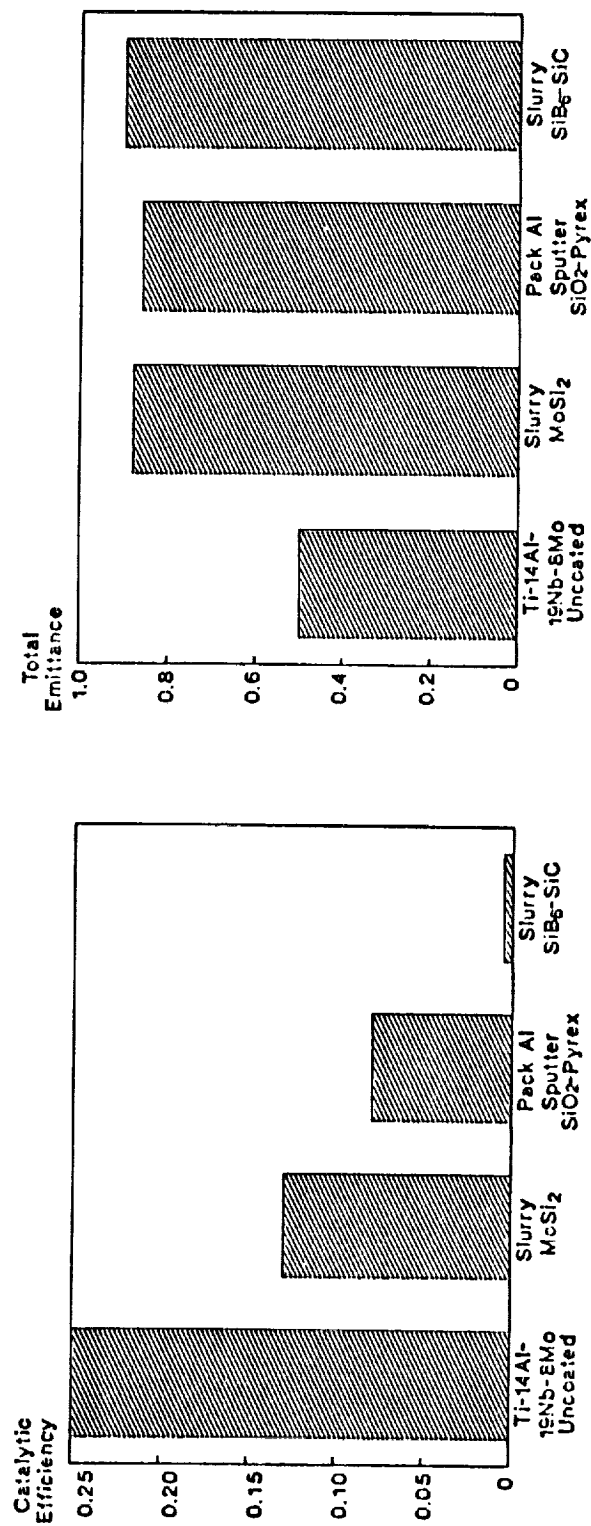


Figure 38(b).

COATINGS IMPROVE PERFORMANCE OF TITANIUM-ALUMINIDE

Static Oxidation: 1 Hr 1800°F

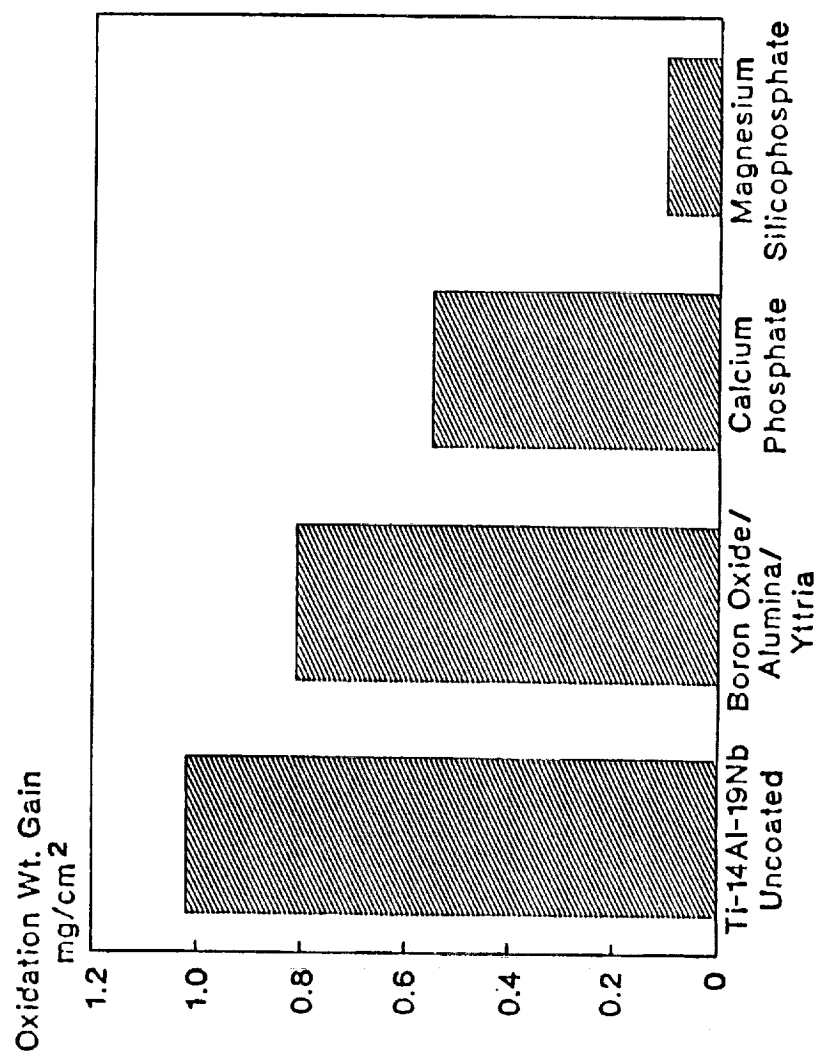


Figure 38(c).

HIGH TEMPERATURE ALUMINUM ALLOYS FOR HEAT SINK TANK STRUCTURE

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Ext. 43135 April 1989
RTOP 505-63-01
Code RM WBS 52-3

Research Objective: Develop high temperature aluminum alloys and associated processing practices for application with heat-sink flyback booster concepts for minimum weight and cost of reusable launch vehicles.

Approach: The research focused on rapidly solidified, powder metallurgy Al-Fe-V-Si system with controlled amounts of strengthening dispersoids and on specific ingot metallurgy Al-Li alloys (Weldalite™). The performance of these emerging alloy systems was evaluated at temperatures typical of those expected on heat-sink tank structures. Alloy chemistry and thermal-mechanical processing was modified as required to obtain the best combination of mechanical properties and processability.

Accomplishment Description: The mechanical properties of several Al-Fe-V-Si alloys and Weldalite™ have been determined from room temperature to 600°F. Both alloy systems have specific strengths and moduli significantly greater than those of a state-of-the-art 2219-T87 aluminum alloy that is currently used on the external tanks of the shuttle launch system (see figure). The Al-Fe-V-Si alloy having the best properties had a composition that produced the highest dispersoid content (37 v/o). This alloy shows high strength and superior stiffness up to 600°F. The Weldalite™ material has superior strength up to about 500°F and good stiffness over the temperature range investigated. Based on current projected trajectories for an advanced launch system, temperatures on a heat sink tank wall are expected to be between 400°F and 600°F. Therefore both the rapidly solidified Al-Fe-V-Si alloy and the ingot alloy Weldalite™ appear to be prime candidates for application on such a vehicle.

Significance: Results to date show that the candidate high temperature aluminum alloys exhibit the properties necessary to be utilized as material for heat-sink tank applications.

Future Plans: Research will be expanded to further characterize these candidate alloys and processing techniques and to evaluate small test subcomponents incorporating tank structures and insulation systems.

Figure 39(a).

SPECIFIC STRENGTH AND STIFFNESS OF CANDIDATE HIGH TEMPERATURE ALUMINUM ALLOYS

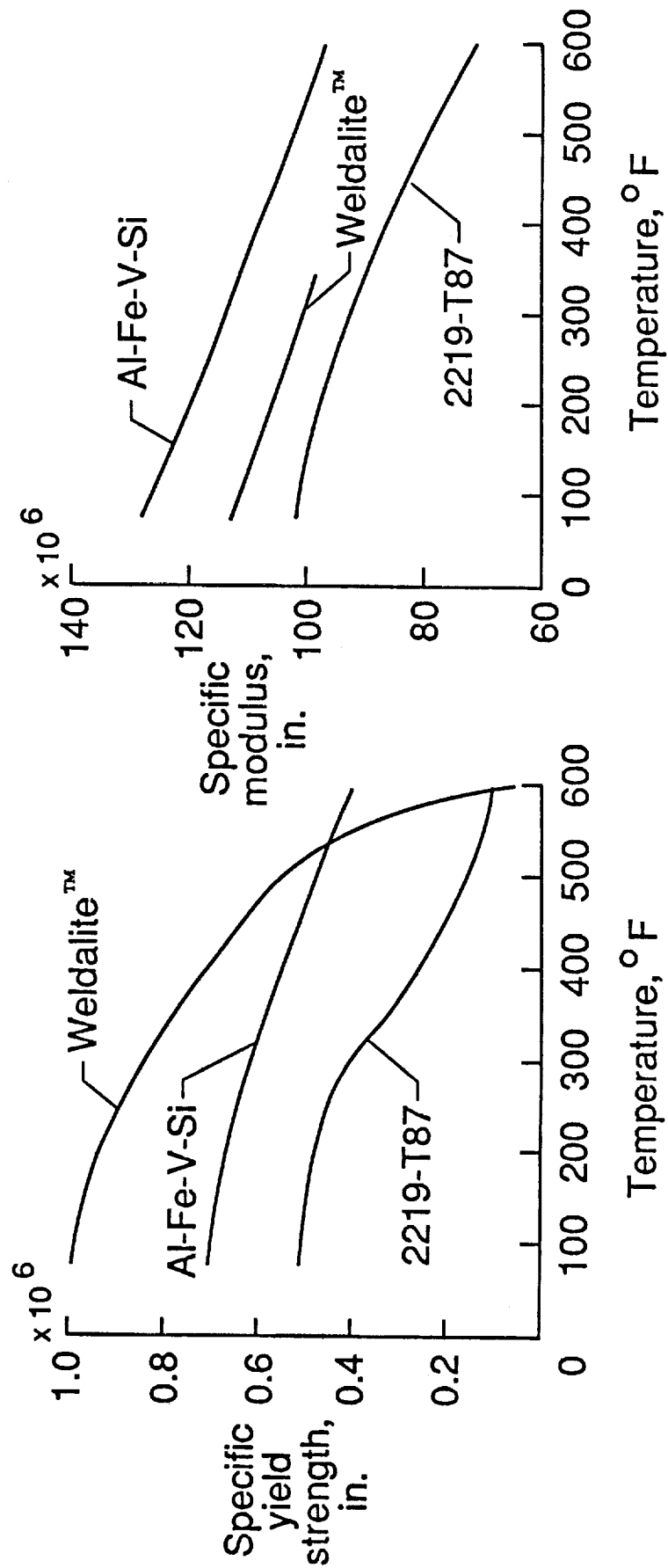


Figure 39(b).

DEVELOPMENT OF TITANIUM BASED METAL MATRIX COMPOSITES
FOR HIGH TEMPERATURE HYPERSONIC APPLICATIONS

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Ext. 43512 June 1989
RTOP 763-01-41
Code RM WBS 54-4

Research Objective: Investigate fiber reinforced metal matrix composites for hypersonic vehicle structures and develop unique fabrication and processing techniques to optimize property combinations.

Approach: Conduct a comprehensive analytical/experimental program to characterize the effects of simulated service environment on the mechanical properties and microstructural stability of fiber reinforced titanium alloy matrix composites. Develop consolidation and processing techniques to optimize properties and stability at expected service temperatures.

Accomplishment Description: In an investigation at LaRC, the mechanical properties of Ti-14Al-21Nb (Ti₃Al based) and Ti-15V-3Al-3Cr-3Sn (beta titanium based) alloys reinforced with SCS-6 silicon carbide fibers have been measured at room and elevated temperatures after various processing and mission simulation cycles. The figure shows the modulus of these two alloys in the reinforced and unreinforced condition as a function of temperature. The unreinforced Ti-14Al-21Nb and Ti-15V-3Al-3Cr-3Sn specimens were fabricated from material which was exposed to simulated composite consolidation time-temperature profiles. Both of the reinforced alloys contain about thirty volume percent fibers oriented parallel to the loading direction (unidirectional) and exhibit vast improvements in modulus over the unreinforced alloys at all temperatures examined. The processing conditions for both of the composites have not been optimized as yet, but these limited data illustrate the potential of titanium matrix composites for much higher temperature applications than unreinforced titanium alloys.

Significance: Future generation hypersonic vehicles will require lighter weight and higher temperature structures than current vehicles. They will require the use of low density materials with good high temperature strength and stiffness. Fiber reinforced titanium alloy matrix composites have the potential of meeting these needs. The results of this study clearly demonstrate that titanium matrix composites have potential for contributing to significantly lower weight in hot structures of hypersonic vehicles.

Future Plans: Determine optimum consolidation parameters for fabrication of composites with various titanium alloy matrices, alternate reinforcing fibers, and varying fiber orientations. Determine high temperature mechanical property behavior of these new systems after

Figure 40(a).

MODULUS BEHAVIOR OF TITANIUM BASED METAL MATRIX COMPOSITES

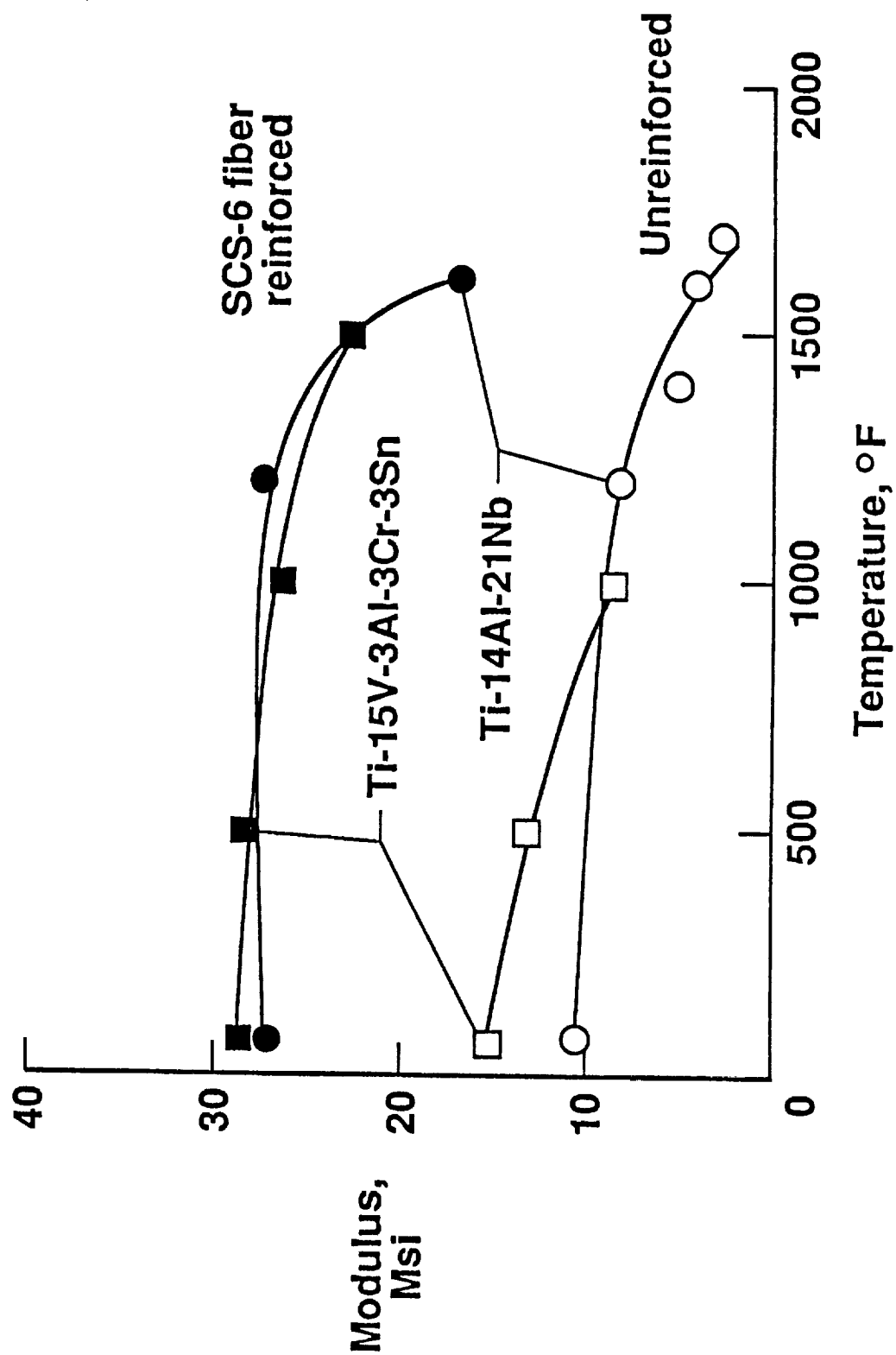


Figure 40(b).

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